THE JOURNAL

OF THE

AMERICAN LEATHER CHEMISTS ASSOCIATION

VOL. XLVI AUGUST 1951 No. 8

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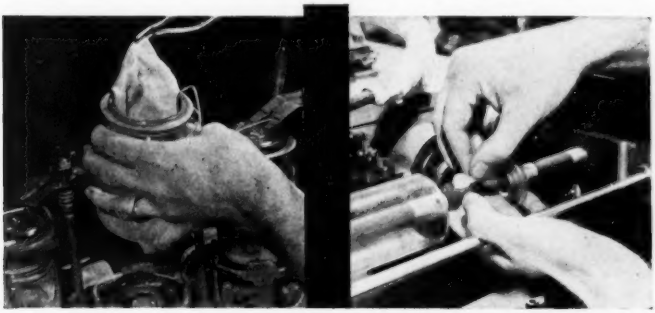

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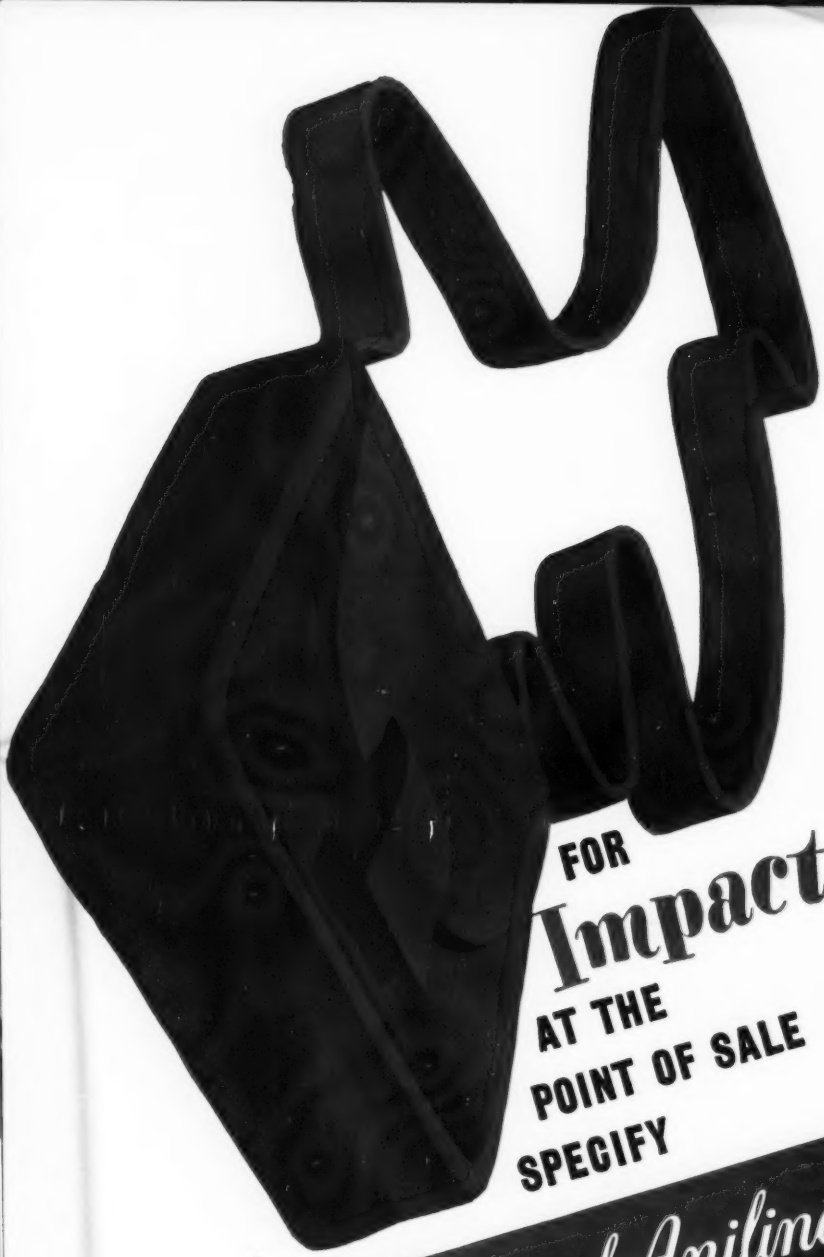

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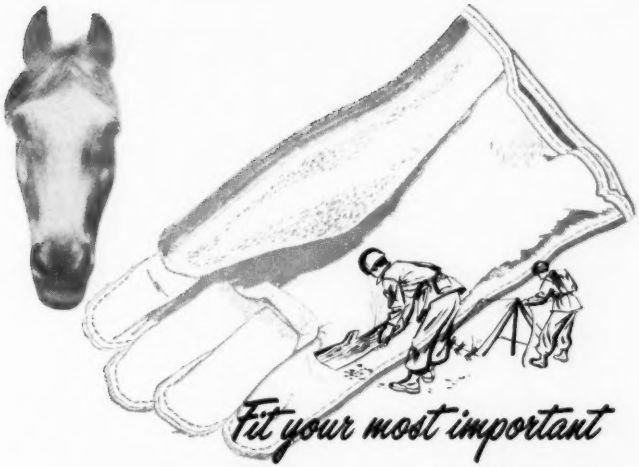

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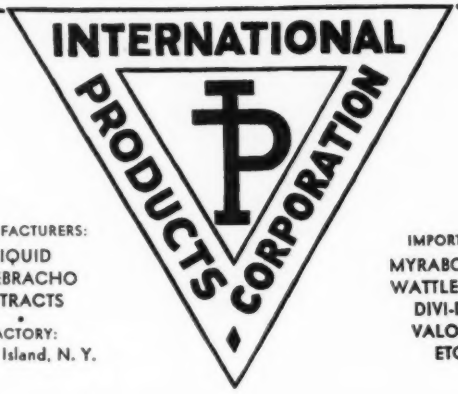

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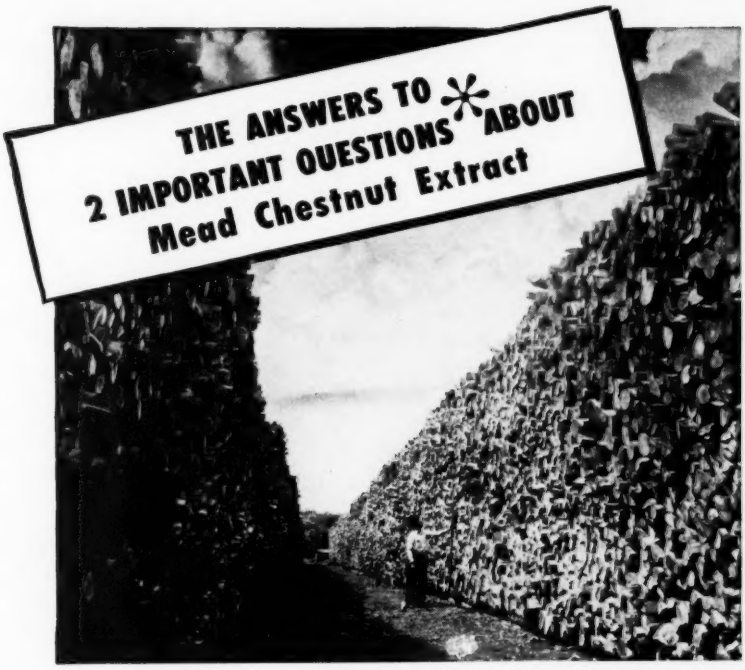

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Tanning Material A	61%
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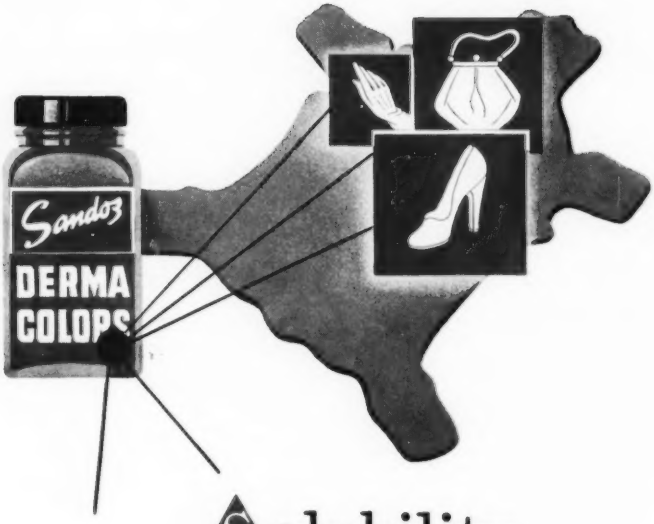

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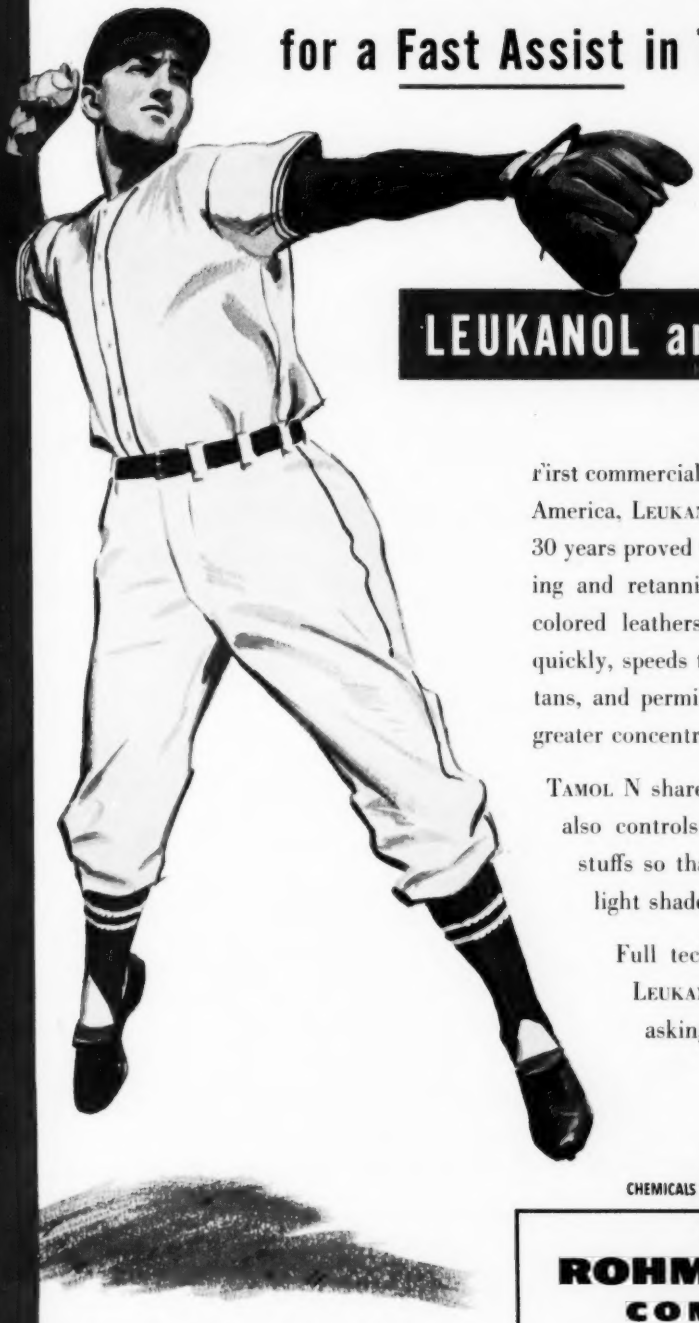

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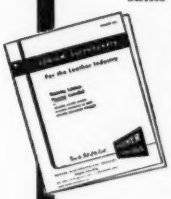

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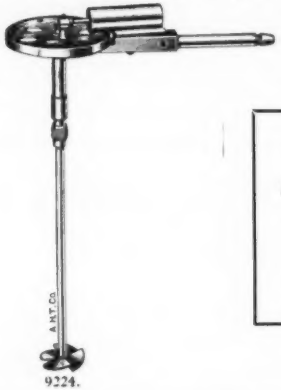

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THE FORTY-SEVENTH ANNUAL MEETING

The Forty-Seventh Annual Meeting, held at the Griswold, Groton, Connecticut, from June 11 to 13, was one of the most successful meetings in the history of the Association. Close to 500 persons, of whom about 100 were women, were in attendance.

On June 10 Committee meetings were held, thus disposing of this important feature of Association work before the regular sessions began.

Due to the excellent organizational work of the program committee under the leadership of Ed Flinn, just enough papers were accepted so as to allow for their unhurried presentation and discussion. They were of sufficiently diversified nature to stimulate interest at every session. Outstanding in interest, however, was the Symposia on Advances in Leather Technology held Tuesday morning under the panel chairman, E. R. Theis.

The technical sessions extended from Monday morning to Wednesday noon. Wednesday afternoon featured the annual golf tournament, while the annual banquet was held Wednesday evening. At the banquet the first Ade Winheim Memorial Award, given to the leather chemistry student presenting the most outstanding paper contributing to the science of leather chemistry, went to Dr. Shu Tung Tu. The award was given for the paper on "Concept of the Mechanism of Tannages of Phenol Substances". The presentation of the award was made by Mrs. Winheim.

No Alsop Award was made this year.

Councilors elected were Wallace Windus, Robert Hobbs and Charles Morrison.

In the golf tournament, the Teas Memorial Cup went to James Morrison with low gross of 81. The P. F. Reilly Cup was won by J. Pfeiffer with low net of 71. K. Garverick won the Cuthbert Memorial Cup with low net open of 61.

The program of the Annual Meeting follows.

Technical Program

Morning Session, Monday, June 11

R. M. KOPPENHOEFER
Presiding

9:30 A.M.—BUSINESS MEETING AND PRESIDENT'S MESSAGE
by R. M. KOPPENHOEFER
Socony-Vacuum Laboratories

10:30 A.M.—THE FUNCTIONS OF THE LEATHER DIVISION OF THE NATIONAL PRODUCTION AUTHORITY

by E. R. LOBAUGH,
Leather and Leather Products Division, NPA

11:00 A.M.—THE DRY STRENGTH OF COLLAGEN FIBRE BUNDLE AGGREGATES

by W. T. RODDY
Tanners' Council Laboratory
University of Cinncinati
Discussion by E. D. COMPTON

11:30 A.M.—EFFECTS OF TANNERY PROCESSES ON THE ELECTRON MICROSCOPIC APPEARANCE OF BOVINE HIDE COLLAGEN FIBRILS

by R. BORASKY and J. S. ROGERS
Eastern Regional Research Laboratory
Discussion by J. H. HIGHBERGER

Afternoon Session, Monday, June 11

F. O'FLAHERTY
Presiding

2:00 P.M.—THE LEATHER CHEMIST ANALYZES HIS INDUSTRY

by KENNETH E. BELL
A. C. Lawrence Leather Co.

2:45 P.M.—SOME NOTES ON THE MIXING OF TAN YARD AND RETAN LIQUORS USING VARIOUS VEGETABLE EXTRACTS

by SEDGWICK K. JOHNSON
Discussion by T. D. BRAUNSCHWEIG

3:00 P. M.—ENZYME EVALUATION BY VISCOSITY REDUCTION

by D. F. HOLLOWAY
A. C. Lawrence Leather Co.
and R. F. NICKERSON
Monsanto Chemical Co.
Discussion by I. C. SOMERVILLE

3:30 P.M.—PRACTICAL PROCEDURES FOR OBTAINING AND ASSESSING SIGNIFICANT IMPROVEMENTS IN THE THE QUALITY OF INSOLE LEATHER

by S. S. KREMEN
Tanners' Council Laboratory
University of Cincinnati

Discussion by C. G. TELANDER

4:00 P.M.—STUDIES ON THE POLAR AMINO CONTENT OF COLLAGEN AND RELATED MATERIAL

by J. M. CASSEL, E. H. McKENNA and A. C. GLIME
Leather Section, National Bureau of Standards

Discussion by ROBERT STUBBINGS

4:30 P.M.—THE SAMPLING OF SIDE UPPER LEATHER

by C. W. MANN
Textile, Clothing and Footwear Section
Office of the Quartermaster General

Discussion by W. MORRIS

Morning Session, Tuesday, June 12

E. R. THEIS
Panel Chairman

9:00 A.M.-12:00 N.—SYMPOSIA—ADVANCES IN LEATHER TECHNOLOGY

(1) Intelligent Cooperation of Chemist and Production Man . . . ROLF QUARCK
(2) Replacement Tanning Materials . . . . . H. G. TURLEY
(3) Sulfonyl Chlorides as Tanning Agents . . . . . C. H. GEISTER
(4) Iron Tannage Developments . . . . . W. O. DAWSON
(5) Fat Liquoring of Iron Tanned Leathers . . . . . C. E. RETZSCH

DISCUSSION

(6) Practical Uses of G-942 Tanning Agent . . . . . W. K. SCHWEITZER
(7) Zirconium Tannage . . . . . I. C. SOMERVILLE
(8) Politan-resorcinol-formaldehyde Tannage . . . . . W. WINDUS
(9) Replacement Fats . . . . . A. SCHUBERT and H. B. MERRILL

Afternoon Session, Tuesday, June 12

E. B. THORSTENSEN
Presiding

2:00 P.M.—THE LEATHER CHEMIST OF 1951—AN APOLOGY FOR PROF. H. R. PROCTOR

by H. G. TURLEY
Rohm & Haas Company

2:30 P.M.—THE INFLUENCE OF SPLITTING ON THE STRENGTH OF CHROME-TANNED STEER HIDES

by J. R. KANAGY, W. H. LESER, E. B. RANDALL, T. J. CARTER and C. W. MANN
Leather Section, National Bureau of Standards

Discussion by M. MAESER

3:00 P.M.—THE APPLICATION OF STATISTICS IN THE TANNING LABORATORY. PART I. THE USE OF A YOUDEN SQUARE

by F. D. THAYER, JR., E. G. BIANCO and F. WILCOXON
American Cyanamid Co.

Discussion by R. M. LOLLAR

3:30 P.M.—RECENT FINDINGS ON TANNING PROPERTIES OF LIGNOSULFONATES

by M. BAUM, R. LOVIN and J. R. SALVESEN
Marathon Corp.

Discussion by G. W. SCHULZ

4:00 P.M.—A STUDY OF THE FACTORS WHICH INFLUENCE THE ABSORPTION OF LIGNINSULFONATES BY HIDE POWDER

by H. B. MARSHALL, M. KRIZSAN and W. C. HENRY
Ontario Research Foundation

Discussion by F. F. MARSHALL

4:30 P.M.—USE OF POLYSULFIDE LIQUID POLYMERS FOR IMPREGNATION OF LEATHER

by K. R. CRANKER and J. S. JORCZAK
Thiokol Corporation

Discussion by RENE OEHLER

Morning Session, Wednesday, June 13

J. H. HIGHBERGER
Presiding

9:00 A.M.—SOURCES OF VARIANCE IN THE SPECIFICATION PROPERTIES OF ARMY RETAN SHOE UPPER LEATHER

by R. M. LOLLAR
Tanners' Council Research Laboratory
University of Cincinnati

Discussion by F. L. COLLINS

9:30 A.M.—STUDIES IN TWO BATH CHROME TANNING. II. INVESTIGATION OF RELATION BETWEEN CHROMATION AND REDUCTION

by K. KLANFER and B. ROQUETTE

Discussion by L. SELIGSBERGER

10:00 A.M.—STUDIES IN CHROME LIQUORS XV

by T. C. THORSTENSEN and E. R. THEIS
Lehigh University

Discussion by R. S. ADAMS

10:30 A.M.—FATLIQUOR DISTRIBUTION IN CHROME-VEGETABLE RETANNED SIDE LEATHER

by B. ROLL
Moench Leather Company

Discussion by B. A. GROTA

11:00 A.M.—ALUMINUM ACETATE IN DEVELOPMENT OF ALUM RETANNAGE

by W. F. HAPPICH, C. W. BEEBE and J. S. ROGERS
Eastern Regional Research Laboratory

Discussion by G. A. BUTZ

11:30 A.M.—SOME FORMATE CHEMISTRY AND ITS APPLICATION TO THE PICKLING AND TANNING OF CHROME LEATHER

by J. W. HARNLY and J. R. RODDEN
A. H. Ross & Sons Co.

Discussion by J. H. STUMPF

THE PRESIDENT'S MESSAGE

By R. M. KOPPENHOEFER

For many years now we have conducted no "business meeting" during our annual session. This year, as you will note from the program, the first hour has been given over to certain important matters upon which the membership must take action. I am going to preface this "business meeting" with a few preliminary remarks.

It is not my purpose, at this time, to discuss with you any of the technical aspects of the leather industry or the manner in which the technical representatives of the industry can more effectively solve many of the problems with which we are now faced. Rather, at this time, I am going to attempt to make you more familiar with your Association and with the affairs of the Association over the past year.

I took occasion the other evening to review the Journals of ten years ago and to note the growth of the Association since our meeting in Columbus in 1941. At that time, we totalled 494 members., of which 235 were active, 211 associate and 48 mutual. Our most recent accounting lists 957 members, representing 47 states and 27 foreign countries. Thus in the last ten years, our membership has increased 100 per cent. This growth, in spite of the difficulties which the industry has faced from aggressive competition and from artificial economic factors, is truly remarkable. In the matter of finances, our position has been likewise improved. Our income in 1950, as you may have read in the Journal, from both the General Account and the Journal was $27,090. Our expenses during the past year were $25,838, leaving a net to surplus of $1,252. Comparing this with 1941 when our income was $15,421 and our expenses $13,433, you will note that not only in membership have we progressed but in ability to transact our affairs. Our 1951 budget lists a tentative income of $26,378 and prospective expenses of $26,275, leaving a net to surplus of $3.00. Our present surplus or net assets are $37,225 compared with $8,870 ten years ago.

Before going into the reasons for presenting this information, I would like to say that these results attest to a policy adopted ten years ago of turning over our business affairs to those members skilled through experience in this activity. It attests to the hard work of Mr. Schiller and Mr. Lincoln and to the present Chairman of the Ways and Means Committee, Mr. Teas. Our Association, I might venture to say, is perhaps alone among technical societies in that we have not made any changes in our assessments or dues to members for the past 25 years. Our expenses of operation of the Secretary's Office, and particularly in the Journal have increased as the value of the dollar decreased and the membership became larger.

Today, we are not recovering from our dues the cost of publication of the Journal, which is now somewhat over $1.00 per copy. Certain groups have

legitimately found it expedient to join the Association as associate members in order to save on the Journal subscription. This fact, plus the fact that our operations during this coming year may represent a deficit, has prompted Council to consider means of increasing revenues. If the new By-Laws are approved, it is the intention of Council to act on a $2.00 per year increase in dues. We believe this modest increase will permit continued operation at present levels.

Our growth, technically, has been equally impressive. Through the ravages of war, the activities of many of our associate societies in foreign countries have diminished and our Journal has assumed an ever increasing importance in the field of leather technology. Our circulation, at present, is 1,363 and the Journal goes to practically every country in the world, including a few behind the Iron Curtain. The printing of the Journal represents the major expenditure of our operation and Council has taken five steps in the past year designed to curtail operating expense in this respect without, in any way, affecting the quality of the publication. These are as follows:

(1) In the future, reprints purchased from the Association will be supplied at cost plus a 25 per cent additional charge to cover the cost of handling. Many times these reprints are used for advertising purposes and it was the feeling of Council that the Association should not bear the additional expense which is always incurred in handling such items.

(2) The price for foreign subscriptions will be increased $1.50 over that of American and Canadian subscriptions. This move, in line with action taken by other technical societies, will recover for the Association the added mailing cost involved in supplying copies to foreign countries.

(3) A study is again being made of the quality of the paper which is being used in our Journal to determine if a satisfactory paper of lower cost might not be made available.

(4) Within the next six months, we will review with our Ways and Means Chairman our present advertising rates which again have not been changed for many years. Our policy, in this respect, as compared to other technical and trade papers, is again unique.

(5) Consideration was given, over the past year, to the petition from our membership to bring our Cumulative Index of the Journal up-to-date. This would mean compiling a record of all publications since 1925 and represents a really tremendous assignment, and one which would have to be performed by outside help since it could not be handled by the present staff of the Journal. Mr. Williams has estimated that the cost for compiling a 10 year Index would be $15,000 and it is thus evident that the bringing of the Index up-to-date would completely wipe out our present reserve. We have discussed this matter with the leading research organizations of our industry and with many of our leading members, including those who have worked on the business end of this organization. The prevailing opinion was that this should not be done.

We attempted in January to obtain some indication from our membership as to whether they would be willing to purchase such an Index at a fraction of the cost of its production. The response was preponderantly negative. In light of this, Council has taken no action on this petition and if action is to be taken it will require a more whole-hearted support of the membership than has been obtained to date.

Passing on to what I regard as the the work of our technical committees, I would like to say that, in this respect, we have not progressed nor has the same spirit been shown by our membership as a whole. The strength of the organization, the very purpose for its existence, lies in the progress of the work of these technical committees. In other technical societies, with which I have been associated, the membership, at large, has supported these technical activities with enthusiasm, such that the obtaining of the co-operative work which is necessary has never been a task. On the other hand, in my association with the organization, I can recall no single instance of any member having asked to serve on the technical committees which we have in existence.

Our membership has come rather by solicitation and request. I should like to ask you to give this point careful consideration, to review our present list of committees and to determine for yourselves whether you can help for I feel, through mutual participation in this type of activity, our industry, as well as our science, is materially assisted. Our present committees are headed by the most able men of our organization, but too much of the work has been confined to too few.

I call attention, at this time, to the very important problem of the Hide Powder Group and the matter of sustaining, in the face of present conditions, a continued supply of the basic commodity standard hide powder. I call attention to the activity of the Physical Testing Group, who, at present, are re-writing and re-editing all of our physical test methods on which so much work has been done in recent years. I also call attention to the new Committee which has been formed on Leather Finishes for the purpose of supplying standard techniques of analyses for this important group of commodities.

The Association, through your Council, has also elected to participate in the International Union of Leather Chemists which was formed in Europe. We have participated in this activity because we felt it was an obligation to further technical progress of the industry. We are not convinced that, because of the distances involved, we can be of assistance and that such participation can be helpful. We are, however, continuing in this preliminary stage until a more intelligible decision can be made in this matter.

I have thus reviewed the major activities of the Association over the past year. Our year began in June with a series of unfortunate events. The first Sunday in June, when I was advised of Ade Winheim's tragic death returning

from our Convention and the critical injury to his wife, I was shocked and felt a very deep personal loss. This was followed, a week later, by the death of Ted Oberlander, with whom I had worked quite closely in recent years and who had served as President preceding Mr. Winheim. Later in the year, the deaths of Ed Cavett, who was well known and who had worked hard in the Association, and John Potts, who was one of our most practical members, also represented a distinct loss. I cannot pass at this time without paying respect to these men.

At the November meeting of Council, resolutions were passed and spread upon the minutes commemorating the services of Mr. Winheim and Mr. Oberlander, both past-presidents. Because of the unusual circumstances surrounding Mr. Winheim's death and because he was foremost in bringing to our Association modern approaches to the operation of technical societies, Council felt that some additional commemoration should be taken. The Winheim Award was established, as a result of Council action, to be given biennially to the student who presents the best paper in the field of leather chemistry. The first award of the Winheim Medal will appropriately be made later in this meeting. With this resume of the affairs of the Association, I now pass on to the important business meeting.

X-Ray Diffraction Studies

A Review of Recent Researches which Concern Collagen

by RICHARD S. BEAR

Department of Biology
Massachusetts Institute of Technology

The purpose of this review is to bring to the attention of the tanning and leather industry a series of papers concerning the fine structure of collagen fibrils, as deduced from X-ray diffraction studies by R. S. Bear and O. E. A. Bolduan, with the collaboration of T. P. Salo in chemical matters. This work, in close connection with similar electron microscope studies of F. O. Schmitt and J. Gross, was carried out in the Biology Department of the Massachusetts Institute of Technology as a part of a research program sponsored by the Office of the Quartermaster General, Research and Development Branch, under a special project 130-46, Determination of the Values and Properties of Skin Structure. The project was conducted under the administration of the Leather Subcommittee of the National Research Council on Quartermaster Problems.

Historical

Crystallography has been an important scientific field for several centuries. Sometime after the discovery of X-rays by Rontgen in 1895, it was shown, notably by Friedrich and Knipping, von Laue and the Braggs (1912-13), that this electromagnetic radiation of extremely short wave length could be used to determine the positions of atoms and molecules in crystalline structures.

Early in the 1920's, largely due to the efforts of Herzog and collaborators, it was found that many animal and plant tissue components, particularly fibrous structures, have a regular construction much like that of crystals. When a fiber is placed in a narrow beam of X-rays, its regular arrangement of molecules produces a pattern of deflected (diffracted) X-ray beams which can be registered on a photographic film. Analysis of the pattern observed tells how the atomic and molecular components of the fiber are organized.

Diffraction studies of gelatin and collagen first appeared in 1920–1926. Early investigations were those of Scherrer (1920), Herzog et al (1924–1926), Katz and Gerngross (1925, 1926a), and Heringa and Kolkmeijer (1926), who established the general nature of collagen diffraction patterns and the similarities between the patterns of collagens and gelatins from a number of sources.

It was, of course, almost immediately recognized that the diffraction method might be useful in investigating the alterations to the structure of the collagen fibers of skins and hides brought about by tanning operations. Outstanding early application of this sort was by Katz and Gerngross (1926b, 1931). Subsequent notable contributions are those of Küntzel (1936), Lloyd (1935, 1938), Highberger and Kersten (1938, 1939), and Astbury (1940).

For a foundation upon which to base conceptions of tanning action one requires models of collagen fibril structure, which should be derivable from X-ray diffraction studies. Early work distinguished two types of structure, that within and between micelles. The micelles are local regions in which there is fair, almost crystalline arrangement of the polypeptide chains. Gerngross, Herrmann and Abitz (1930) pictured the micelles as being fringed with segments of chain which serve to bind or entangle the micelles into a continuous structure. The intermicellar portions, at which the fringes join the micelles, is less regular or amorphous in organization.

Herzog (1927) suggested that tanning agents penetrate the intermicellar locations preferentially. Since it is the micelles that give rise to the more characteristic aspects of the (wide-angle) X-ray diffraction observed in the early studies, it is not surprising that most investigators have not found distinctive alteration of the diffraction pattern to result from tanning, although penetration of tanning material eventually causes considerable deterioration of the diffraction diagram.

Astbury (1940) has been most active in considering the configuration of collagen polypeptide chains, and his views have received wide distribution. The models developed up to this point may, however, now be regarded as classical, since they preceded the application of the more recently developed techniques of small-angle X-ray diffraction and electron microscopy, which have yielded evidence requiring radical modification of ideas concerning collagen fine structure.

The laboratories of Clark (1935) and of Wyckoff (1935) simultaneously discovered that prior diffraction studies had missed certain data observable very close to the undiffracted X-ray beam at small diffraction angles. These diffractions were resolved by Bear (1942, 1944) and by Kratky and Sekora (1943), who showed that collagen fibrils possess a large structure periodically repeated every 640 A (dry case) along the fibril axis. Almost simultaneously, electron microscope studies of Schmitt, Hall and Jakus (1942) and of Wolpers (1943) furnished direct photographs of a banded structure of this size along isolated collagen fibrils.

In the years intervening between 1944 and 1949 the small-angle X-ray diffraction studies were intensively prosecuted, with occasional oral reports before the American Leather Chemists Association constituting the only publicly available presentation of the results. Since then a series of eight papers have appeared in which these studies are described. A summary of pertinent conclusions now follows:

Development of Experimental and Mathematical Methods

As the detailed examination of the small-angle diffraction by collagen progressed, it became necessary to develop methods for investigation in this field to a point hitherto not attempted. The various diffraction spots or lines seen in the diffraction patterns required extreme collimation of the X-rays, such that all of these incident upon an examined specimen would be rendered parallel within about four minutes of arc. X-ray sources are not very intense and their energy disposition is not easily controlled by focusing, so that the problem of photographing the small-angle patterns in reasonable times (up to a few hundred hours) is severe. No systematic treatment of the problem of efficiently defining narrow beams with the desired qualities had previously appeared

This problem was considered by Bolduan and Bear (1949), who prepared cameras especially suited for the various tasks relative to the collagen investigation. Complete description of the cameras is given in the cited reference, along with samples of results achieved and a general consideration of camera design requirements.

As the experimental development proceeded it became apparent that collagen small-angle diffraction consists of a single related set of lines or spots derived from the fundamental large structural period of about 640 A.

In other words, collagen fibrils gave no evidence of more than one large periodic structural spacing, being in this respect deficient relative to common three-dimensional crystals, which yield three such fundamental spacings (lattice translations). Furthermore, the individual collagen diffraction spots showed characteristic dimensions, shapes, and other qualities which indicated that the collagen fibrils possess hitherto unencountered types of one-dimensional structure.

A program was thereby set up wherein alternate theoretical calculation and experimental tests of the theoretical results eventually narrowed the variety of structural model needed to be considered as applying to collagen.

Certain qualitative and semi-quantitative consequences expected in diffraction by systems 'deficient in order', i.e., lacking the full three-dimensional regularity of normal crystals, were systematically canvassed (Bear and Polduan, 1950a) and used to indicate clearly that collagen fibrils are indeed order deficient (Bolduan and Bear, 1950). More quantitative approaches started with calculation of detailed diffraction characteristics expected of simple cylindrical bodies with periodic structure distributed along the cylinder axes (Bear and Bolduan, 1950b). Then followed a comparison between theoretical and experimental diffraction line shapes and other characteristics (Bolduan and Bear, 1951), which indicated that the actual collagen fibril (dry) shows a mixture of characteristics: it is approximately a simple cylinder, but in addition to the periodic variations in density (chemical composition) occurring along fibril axes there are also periodic variations in diameter (corrugation). Also, there is a tendency for longitudinal cleavage into thinner filamentous subunits which become somewhat displaced and distorted relative to each other within the fibril.

The final coalescense of all of these features into a single structure, capable of accounting for the pertinent characteristics of small-angle diffraction by collagen, was described in a mathematical paper by Bear and Bolduan (1951). More readable accounts, along with application of the results to a variety of situations of more direct practical interest, appeared almost simultaneously. These concluding papers of the series may now be briefly summarized.

The Structure of the Collagen Fibril

The fibril is the characteristic ultramicroscopic element of the histologically visible fibers or fiber bundles of connective tissues, skin dermis and tendons. Each individual fibril consists of parallel 'protofibrils' or polypeptide chains which tend to match their common pattern of chemical features transversely to the direction of their long axes. Figure 1 was used by Bear, Bolduan and Salo (1951) to indicate this type of structure diagrammatically, as well as to show which types of structural feature correspond to which type of diffraction effect at small angles.

At certain levels along the fibril the protofibrils are distorted from perfect packing. These levels correspond to the bands seen as cross striations in

electron micrographs. Existing diffraction and electron optical evidence seems to be in agreement if the *bands* are taken to be positions at which fibril diameters are greater than average, and where the distortion permits an openness of structure at which stain or a tanning agent penetration may occur. *Interband* levels possess a more compact structure which approaches that of crystalline regularity.

By studying the effect of graded sorption of phosphotungstic acid it was possible to show a sequence of events such as would be expected if this stain enters bands preferentially. At high stain uptake the band structure is so badly distorted that the diffraction effects which were used to establish the nature of band level structure are no longer seen, and the axial large period is reduced below 600 A.

The structure at bands is essentially statistical, i. e., corresponding band levels along the fibril are not identical, only averaging alike because of the random nature of the polypeptide chain kinking. Kangaroo tendons, which had been selected for these studies, because of stability and good orientation of constituent fibrils, were found to exhibit particularly well the diffraction phenomena which make the nature of band distortion clear.

Because all protofibrils or polypeptide chains of the fibril are similarly directed and matched, the pattern of bands and interbands repeated along the fibril is essentially a direct indication, however poorly resolved at present, of the sequence of amino acid residues along individual polypeptide chains of collagen molecules. Bolduan, Salo and Bear (1951) reported results of preliminary attempts to tag specific residue side chains. Most significant results were obtained with deaminated specimens, from which had been removed predominantly the lysine ϵ - amino groups. Hexametaphosphate (Calgon) served as a tag for amino groups before and after deamination. The diffraction alterations observed were characteristic and possibly indicative of a location of the lysine side chains at band levels.

Other tanning agents, such as formalin, chrome and quebracho showed distinctive alterations of the small-angle diffraction. Because these agents most influence certain of the diffraction lines expected to be most intimately related to the band structure as observed electron optically, the diffraction results are consistent with predominant entry of the tanning agents into band levels.

Most of the small-angle diffraction examinations relative to the effects of added stains or tanning agents must be done with dry specimens, because when water is added it enters both bands and interbands in such large amounts as to overcome largely the relatively minor effects due to the protein and other solid components. At the same time the water smooths out the "kinks" at band levels to some degree, causing a lengthening of the large fibrillar period to around 670 A.

The small-angle studies show clearly that water, stains and tanning agents enter collagen fibrils to affect their structure profoundly. At the same

time, wide-angle diffraction examinations of the same examples failed to disclose any marked change caused by these agents, with the exception of water. It is, therefore, clear that only water, of the materials examined, is able to penetrate the interband levels of the fibril to a marked degree.

One thus arrives at a revised statement of the view of Herzog (see above) relative to the penetration of collagen by tanning agents. The modern fibrillar band levels correspond to part of the intermicellar locations of the earlier investigators, and it is largely here that tanning agents accomplish estabilization of collagen fibrils. The interbands are compact and not readily invaded, except by water; they correspond to the micelles of the older terminolgy and give rise to the wide-angle diffraction studied classically.

It is clear that insofar as the effects of tanning agents on the structure of collagen fibrils are of interest to leather technology, the X-ray diffraction method can contribute most effectively through considerations based upon the hitherto little explored small-angle diffractions. The initial investigations of this relatively new field have not begun to exhaust the possibilities but are of significance at the moment chiefly in indicating the way in which the powerful diffraction method, previously disappointing in its contributions to tanning concepts, can be of future service.

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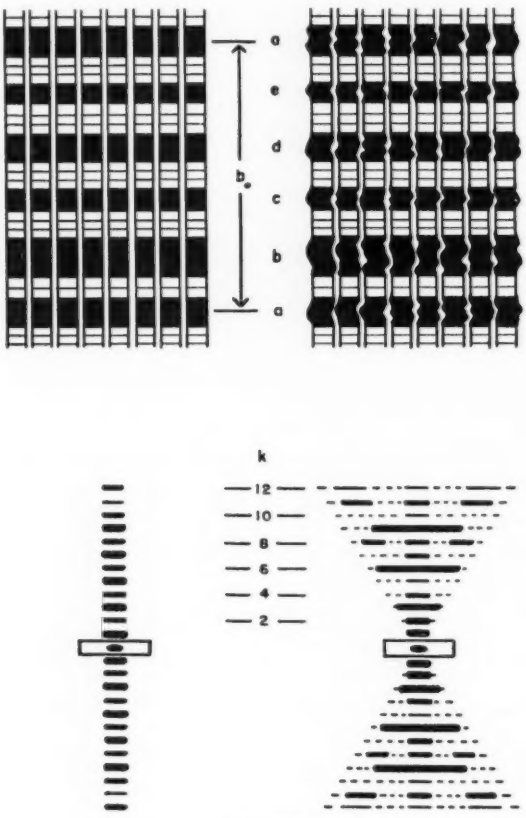

FIGURE 1

The Figure shows longitudinal sections through a 'perfect' model (upper left) for the collagen fibril, representative of its condition when moist, and through a model (upper right) in which some levels are 'imperfect' because of distortions introduced by drying. Eight fine filaments or protofibrils, believed to be essentially individual polypeptide chains, are shown in each case, with the scale for filament thickness exaggerated relative to that for b_o, the axial period. Changes of this period and of interfilament separation with variation of hydration are not shown, but both enlarge (the period goes from 640 to 670 A. and the separation from 10 – 12 to 15 – 17 A.) when a dry specimen is moistened.

The pattern of solid and shaded levels along each filament represents the sequence of chemical structure which is repeated vertically many times in each model, only one full period being shown. Filaments tend to match corresponding features, the solid levels being those at which matching is imperfect and distorted, corresponding to the conditions at the electron optically visible bands, *a* through *e*. The intermediate interbands are shaded horizontally; at these levels structure is relatively compact and well enough organized to yield the small structural details in evidence of wide diffraction angles.

Small-angle X-ray diffraction relates to the over-all large structure of the fibrils. Below each model is shown a sketch of the small-angle diffraction o' served. Perfect fibrillar levels diffract to the short lines or central line components, shown at all lines on the left and at some of them on the right. Imperfection shows up in the progressive lengthening and other details, which become more pronounced as the index, k, numbering the lines increases.

Received May 17, 1951

A Study of Certain Conditions Controlling Wet-Salting of Heavy Abattoir Hides

By C. C. Kritzinger

Introduction

During recent years considerable attention has been given to the problem of setting standards for leather and leather goods manufactured in South Africa. Since the quality of such leathers and leather goods is intimately connected with the quality of the raw material it is quite natural that the possibility of standards for hides and skins should come into the picture.

The quality of a hide is governed by the standard of production up to the time of curing on the one hand, and by the standard of curing itself on the other. Only the latter factor has been considered in the present study. Most of the principles underlying curing have been worked out during the past thirty years, but very few studies have been made on sufficiently large scale to examine the subject from the purely practical point of view.

Experimental

With the co-operation of the Consolidated Hide and Skin Traders' Ltd., the Livestock and Meat Industries Control Board, Silverton Tannery Ltd, and Bagshaw, Gibaud & Co. a series of large scale experiments was planned to give information on the following factors involved in the curing of heavy hides:

1. The amount of salt applied,
2. The use of sodium fluosilicate (silicofluoride) as an additive to the curing salt,
3. The length of the curing period,
4. The shrinkage during curing,
5. The loss in weight during transit,
6. The yield of limed stock,
7. The analytical values for moisture, sodium chloride, water-soluble nitrogen and volatile nitrogen.

In each experiment 30 green hides were used, each hide being washed and weighed after 45 minutes of draining to determine the amount of salt required. The salt was spread as evenly as possible, by hand. Where sodium fluosilicate was used, 2 per cent on the salt weight was well mixed with the curing salt. Six separate stacks, in three pairs, were built with 90 hides in each stack. The lower 30 hides received 100 per cent salt on weight, the middle 30 received 50 per cent and the top 30 received 25 per cent. Onto each stack five more hides were put to prevent undue drying of the uppermost hides, and the stacks then covered with damp hessian. Each pair of stacks comprised one stack in which sodium fluosilicate had been added to the salt and a control stack. The three pairs were kept for 10, 30 and 90 days respectively before opening.

When the stacks were opened the salt was shaken off the hides, the hide weight recorded and the stock shipped to the tannery where the received weight was recorded. After normal soaking the hides were limed, fleshed lightly, drained for one hour and the limed weight recorded. In each group of hides the middle 20 were sampled along the backbone, six inches from the tailroot, for analysis before the hides were shipped to the tannery.

The sample strips of about 2 x 4 inches, were cut into cubes after scraping off all excess salt, the cubes well mixed and divided into three equal portions for triplicate analyses.

Moisture was determined by drying a sample of about 5 grams for 16 – 18 hours at 98 – 100°C.

Sodium chloride was determined by a method described earlier [1]. A sample of the cubes is digested under reflux with NaOH, the solution acidified with nitric acid and a slight excess of silver nitrate added to an aliquot. The excess silver nitrate is titrated with KCNS in the presence of a ferric alum indicator.

Total extractable nitrogen was determined by milling 50 grams of cubes in 250 ml. of distilled water for three hours at 60 r.p.m. This time is not sufficient to bring the extractable nitrogen to equilibrium but is sufficient for the

volatile nitrogen [2]. On a sample of 50 ml. of filtrate (using a cloth) determine the total extractable nitrogen and use a sample of 150 ml. for determining *volatile nitrogen*. Calculate the extractable nitrogen on the total amount of water present (i. e. water added plus water initially present in the cubes) and express as a percentage of the total hide nitrogen. Express volatile nitrogen as a percentage of total hide nitrogen on the same basis.

Examination of Hides

The most outstanding feature of the different stacks of hides was the efficiency of sodium fluosilicate in preventing the development of red heat, except for a few isolated spots. The fluosilicate hides were whiter in appearance and gave the impression of being more moist than the controls. In the control hides those kept for only ten days also did not show red heat, but the 30- and 90-day controls had been severely attacked and had developed a very putrid smell. This was to be expected, since the hides had been in curing during the summer period. The red heat development was more pronounced on the 100 per cent salt hides, while the 25 per cent salt hides showed only traces although they appeared to have suffered most through putrefaction. This observation is in accordance with general experience, viz., that excess salt always shows more red heat development. This is probably due to the fact that the red heat bacteria are aerobic and are supplied with air where excess salt prevents the hides from coming into too close contact with one another.

In all six stacks it was obvious that 25 per cent salt on the hide weight was not sufficient since all the salt had been absorbed or had run away in the form of brine. 50 per cent salt could be sufficient if applied evenly and 100 per cent salt appeared to be excessive. Not all of this excess salt could be shaken off before weighing and bundling. It should be noted that in the case of the fluosilicate hides receiving 100 per cent salt for 10 days a very fine salt was used which clung tenaciously to the stock. Finally, due to circumstances beyond experimental control, the lower hides (i. e. those receiving 100 per cent salt on weight) in the two stacks kept for 90 days became wet, so that the shrinkage recorded was somewhat lower than it should have been.

From the appearance of the hides it seemed that the best cure was obtained when about 50 per cent salt on green weight was used, sodium fluosilicate having been mixed with the salt, and the hides kept for 30 days.

The Results

In the following Tables the results of the various weights and analyses have been set out for the 10–, 30– and 90–day curing respectively. The percentage shrinkage is calculated as the loss on the green weight. Tannery loss is a percentage on the cured weight, but total shrinkage from the green state to the tannery received condition is calculated on green weight. Sodium

TABLE I

Ten-Day Curing

	With Sodium Fluosilicate			Control		
% Salt applied:	25%	50%	100%	25%	50%	100%
Green weight	2002	1998	1997	2243	2120	2203
Cured weight	1624	1676	1754	1783	1716	1856
% Shrinkage	18.9	16.1	12.1	20.5	19.0	15.8
Tannery weight	1619	1649	1692	1764	1714	1823
% Transit loss	.31	.61	3.53	1.06	.17	1.78
% Total shrinkage	19.1	17.4	15.3	21.3	19.2	17.2
Limed pelt weight	1846	1876	1854	2114	1904	2026
% Yield on cured wt.	113.6	111.9	105.7	118.5	110.9	109.1
% Yield on green wt.	92.2	93.9	92.8	94.3	89.8	91.9
% Yileld on Tannery wt.	114.0	113.1	109.6	119.8	111.1	111.1
% Sodium chloride	12.53	12.40	12.24	11.94	12.46	12.29
% Moisture	44.1	36.9	35.6	42.4	37.0	35.9
% NaCl on moisture	28.4	33.6	34.4	28.2	33.6	34.2
% Extract. nitrogen	2.37	2.15	1.89	3.37	2.74	2.62
% Volatile nitrogen	.14	.13	.11	.39	.18	.17

TABLE II

Thirty-Day Curing

	With Sodium Fluosilicate			Control		
% Salt applied	25%	50%	100%	25%	50%	100%
Green weight	2123	1973	2075	2150	2084	2127
Cured weight	1640	1567	1666	1627	1605	1659
% Shrinkage	22.7	20.6	19.7	24.3	22.9	22.0
Tannery weight	1632	1560	1640	1614	1595	1638
% Transit loss	.49	.45	1.56	.80	.62	1.27
% Total shrinkage	23.1	20.9	21.0	24.9	23.4	23.0
Limed pelt weight	2055	1906	2050	2142	2002	2066
% Yield on cured wt.	125.1	121.6	123.0	131.7	124.7	124.6
% Yield on green wt.	96.5	96.6	98.8	99.7	96.1	97.1
% Yield on tannery wt.	125.9	122.1	125.0	132.7	125.5	126.1
% Sodium chloride	11.07	13.43	13.00	12.23	13.30	13.00
% Moisture	38.5	39.9	37.4	42.3	39.4	38.1
% NaCl on moisture	28.8	33.7	34.8	28.9	33.8	34.2
% Extract. nitrogen	2.58	2.33	2.24	3.60	2.77	2.67
% Volatile nitrogen	.14	.13	.12	.62	.33	.26

chloride and moisture figures are for the stock as received out of cure, the concentration of sodium chloride in the residual moisture being calculated. It should again be noted that the method of extracting soluble and volatile

TABLE III

Ninety-Day Curing

% Salt applied	With Sodium Fluosilicate 25%	50%	100%	Control 25%	50%	100%
Green weight	1976	2076	2170	2117	2084	2119
Cured weight	1520	1631	1769	1596	1592	1703
% Shrinkage	23.1	21.4	18.5	24.8	23.6	19.6
Tannery weight	1518	1618	1745	1586	1577	1673
% Transit loss	.30	.80	1.36	.63	.94	1.76
Total shrinkage	23.2	22.1	19.6	25.2	24.3	21.1
Limed pelt weight	2010	2041	2057	2162	1942	2040
% Yield on cured wt.	132.2	125.1	116.3	135.4	122.0	119.8
% Yield on green wt.	101.8	98.3	94.8	102.1	93.2	96.2
% Yield on tannery wt.	132.4	126.1	117.9	136.3	123.1	121.9
% Sodium chloride	13.00	13.47	11.73	13.30	13.23	13.13
% Moisture	41.3	40.0	33.5	42.3	38.7	38.2
% NaCl on moisture	31.5	33.7	35.1	31.5	34.2	34.4
% Extract. nitrogen	2.85	2.58	2.36	4.71	3.06	3.17
% Volatile nitrogen	.14	.14	.12	1.01	.72	.71

nitrogen does not yield all of the extractable nitrogen and in fact would represent only those smaller particles which are easily removable during such a short soaking period [3].

Shrinkage and Transit Loss

In all methods of curing, except perhaps in the case of freezing, the green stock always loses some weight due to the removal of moisture. During sun- and shade-drying the moisture is simply allowed to evaporate but where salt is applied the process is somewhat more involved. Where solid salt is applied to the surface of the hide, as in wet-salting, the moisture on the surface will dissolve the salt slowly to form a brine on the surface. Thus a set of conditions is created where osmosis and diffusion must occur. While sodium chloride from the more concentrated brine on the surface moves into the hide, moisture will move outwards to dilute that brine. Equilibrium is reached when all the residual moisture is saturated with sodium chloride, but in the process a considerable quantity of water has been lost by the hide and has run away in the form of a brine. A similar process occurs during brining. In such curing methods there is furthermore a loss of some nitrogenous compounds, which might materially add to the total shrinkage. The amount or degree of shrinkage measured in practice is therefore the difference between the loss of moisture plus nitrogenous substances and the gain in sodium chloride.

It is obvious that wide variations in the shrinkage figure must be possible. Where the sodium chloride can penetrate rapidly a larger proportion of the moisture will become saturated inside the hide before it has a chance to leave. This is possible, for example, during brining, where fine salt is used or where sufficient excess salt is used. A low shrinkage figure must then result. If hides are stacked in such a way that the moisture cannot run away readily a "heavy" cure will result. Hygroscopic substances such as magnesium or calcium chloride will tend to retain moisture inside the hide where such moisture becomes saturated with sodium chloride to give low shrinkage figures. If the curing process is too short the exchange between sodium chloride and moisture will not be complete, again resulting in less loss of weight. The height of the stack also affects the shrinkage, insofar as low stacks do not exert sufficient pressure to ensure removal of excess moisture. Finally, as demonstrated in Table I for the sodium fluosilicate hides receiving 100 per cent salt, the use of too fine salt which might cling to the stock will result in an apparently low shrinkage.

A "light" cure is caused by the direct opposites of some of the factors mentioned above for the heavy cure. Insufficient salt results in excessive loss of moisture. Coarse salt dissolves too slowly to form a proper brine. Incorrect stacking or too high stacking will lead to excessive seepage of the brine. Too long a curing period may result in partial drying out after the residual moisture has become saturated with sodium chloride, so that a high shrinkage figure is obtained.

The tables reveal that under the conditions of experimentation a shrinkage variation between 24.8 per cent and 12.1 per cent was obtained, depending upon the amount of salt used, the length of the curing period and the presence or absence of sodium fluosilicate. The figure of 12.1 per cent is perhaps excessively low, but it does give an indication of the effect of excess solid salt left on the hides before bundling and weighing.

Loss during transit amounts to less than 2 per cent on the cured weight in all cases except for the one batch discussed already. Here it would seem that part of the value of 3.53 per cent must be ascribed to salt which had shaken off during transit. The train journey was a week in all batches. Since loss in weight during transit may be considered as further shrinkage from the practical point of view, the following discussion will be based upon the total shrinkage figures shown in the Tables and used in Fig. 1 and 2.

Most of the theoretical speculations above are verified by the results obtained. In Fig. 1 and 2 the shrinkage has been plotted against time and against the amount of salt applied respectively. There is a sharp increase in the shrinkage figure for all batches between 10 and 30 days, but continued curing (up to 90 days) does not alter the shrinkage materially. In other words, a period of about 30 days appears to be necessary to obtain equilibrium in the shrinkage. The two batches S100 and C100 (fluosilicate and control

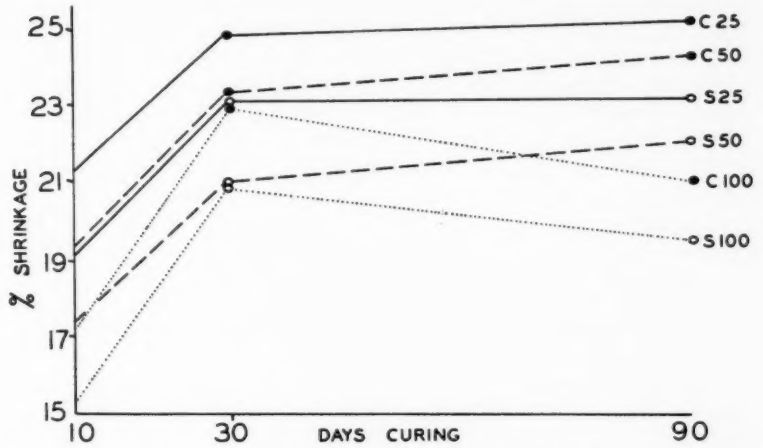

FIGURE 1.—Influence of the curing period on shrinkage of wet-salted hides, using different amounts of salt.

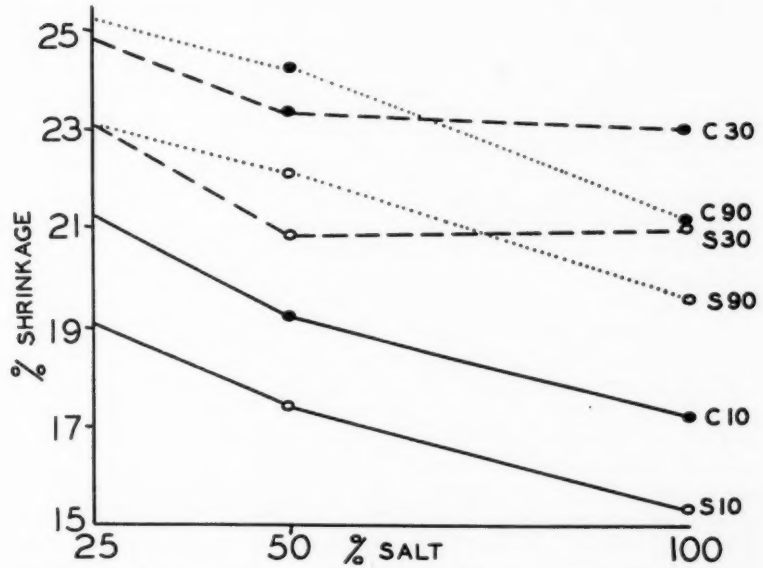

FIGURE 2.—Influence of the amount of salt on shrinkage of wet-salted hides cured for varying periods.

respectively, receiving 100 per cent salt) in Fig. 1 show a decrease after 30 days. This is due to their having been in contact with water shortly before the stacks were pulled, as explained already.

Fig. 2 shows that low shrinkage values will result from the use of larger amounts of salt. The decrease is sharpest between 25 per cent and 50 per cent salt on green weight, and is much smaller from 50 per cent to 100 per cent salt. Once again batches S90 and C90 are out of line, due to the wetting.

It must be clear that time and the amount of salt applied work in opposition to one another. The highest shrinkage value results from the use of too little salt (e.g. 25 per cent) for a long period (e. g. 90 days), and vice versa. The tanner's and the curer's interests therefore seem to clash over the question of shrinkage, but since this is not the only factor concerned in the preservation of hides other factors should be considered too before deciding upon the desired specifications.

From the Tables and the Figures it is evident that the use of sodium fluosilicate as an additive to the curing salt always results in a shrinkage value some 2 per cent lower than the controls, no matter what the conditions of curing. Robertson [4] reported in 1932 that hides cured in New Zealand with salt containing 0.5 per cent and 1 per cent sodium fluoride and fluosilicate appeared to be drier than the controls when they arrived in England. In Australia [5] it was found however that the addition of 1 per cent sodium fluoride to the curing salt resulted in 1.8 per cent less shrinkage and the hides appeared to be wet. A previous study by Kritzinger [6], in which the hides were dipped in a saturated (0.6 per cent) solution of sodium fluosilicate for fifteen minutes before salting, showed that there was so difference in the shrinkage. The number of hides used in the experiment is perhaps subject to criticism, however. Recently [1] it was found that when 3 per cent sodium fluosilicate is mixed with the curing salt the hides retain more moisture but also take up more sodium chloride. No shrinkage figures were taken in that study, but it stands to reason from these findings that the sodium fluosilicate had resulted in a lower shrinkage value. These results have now been fully substantiated.

It might be suggested that the 2 per cent lower shrinkage values of the treated hides can be explained on the basis that sodium fluosilicate is only slightly soluble in saturated sodium chloride solutions, hence the fluosilicate is retained on the hide and leads to low values. This explanation could only hold true in the case of hides receiving 100 per cent salt on hide weight, so that sufficient sodium fluosilicate could be retained. In the case of hides receiving only 25 per cent salt there would not be sufficient fluosilicate to make a difference of 2 per cent in the shrinkage values. However, part of the difference is undoubtedly due to this factor. More important however, is the fact that sodium fluosilicate is a lyotropic agent and would tend to make the hide retain more moisture. This moisture is then saturated with sodium

chloride and a low shrinkage value is obtained. If the hides were dipped in a saturated solution of fluosilicate they would take up only a very small amount of this chemical and the shrinkage would not be affected so markedly.

Once again then, if shrinkage were the only factor to consider in the curing of hides, the use of sodium fluosilicate as an admixture to the curing salt would be welcomed by the curer but condemned by the tanner.

Salt Absorption During Curing

The principles of salt absorption have already been discussed in the above section, and it remains only to study the values in the Tables and the graphs in Fig. 3 and 4. Since the degree of saturation of the moisture with sodium chloride determines the efficiency of salting (see Stuart and Frey [7]) the values of sodium chloride and moisture have been calculated to show the concentration of salt in the moisture retained by the hide after curing.

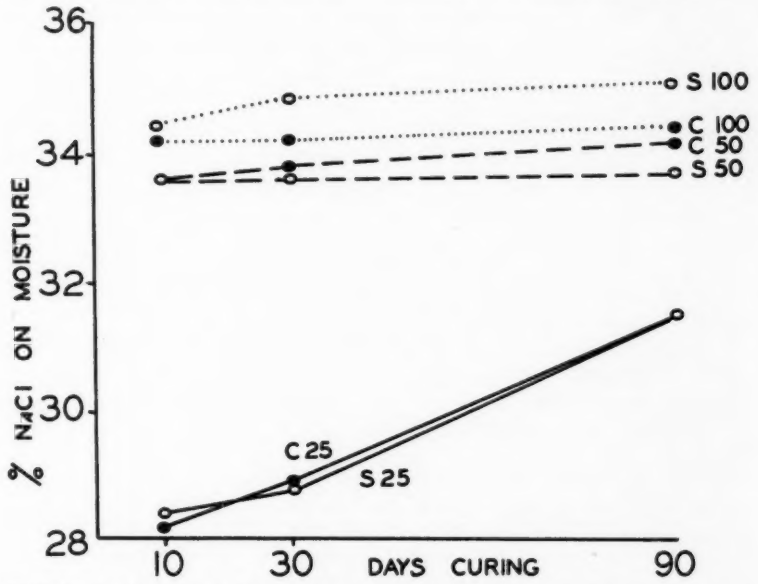

FIGURE 3.—Influence of the curing period on salt absorption in wet-salted hides, using different amounts of salt.

From Fig. 3 it will be observed that as long as 50 per cent or more salt is applied curing for longer than 10 days does not affect the concentration of sodium chloride in the residual moisture to any appreciable extent. In all these cases the concentration is above saturation point (32.6 per cent), indicating that some drying out had occurred after saturation had been reached.

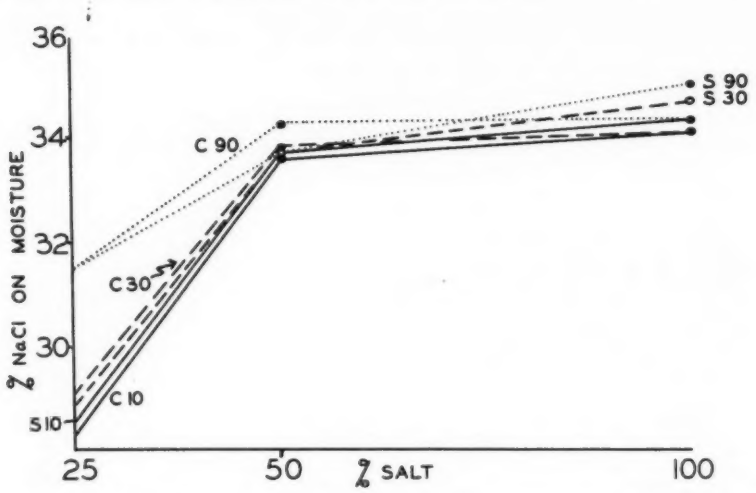

FIGURE 4.—Influence of the amount of salt on salt absorption in wet-salted hides cured for varying periods.

Where only 25 per cent salt on hide weight had been applied the sodium chloride concentration increases steadily as the curing period is increased. This does not mean that more salt goes into solution, since there is no excess salt left on the hide after ten days in cure; the increase is due to slow drying out of the stock. It should be noted that even after 90 days the residual moisture is still not saturated with salt.

It is interesting to observe again that the fluosilicate hides, although they show less shrinkage, have the same sodium chloride concentration (not necessarily the same *content*) as the controls. Furthermore, the 90-day 100 per cent salt hides which had been inadvertently wetted also show full saturation. This means that the retention of moisture or the subsequent up-take of moisture does not affect the final sodium chloride concentration but only the salt content of the stock. The moisture taken up subsequently has merely been saturated again by the excess salt present on the hides.

Effect of Curing Upon Globular Proteins

The globular proteins are subject to a number of changes during wet-salting of hides, all of these changes being due to the presence of salt, to the action of sodium fluosilicate and especially to the activity of putrefactive bacteria which can live in high concentrations of sodium chloride.

When the salt penetrates the hide during the early stages of curing, the dilute brine moving from within the hide to the more concentrated brine on or near the surface will bring with it some of the more readily soluble protein

material. These experiments give no information on the amount of nitrogen lost in this way, but studies by Hausam[8] in 1938 showed that about 1.8 per cent of the total hide nitrogen could be removed during a curing period of about 70 days. Of this amount half was extracted during the first five hours and three-quarters during the first 24 hours. As the salt concentration builds up inside the hide, the globular proteins are salted out and become insoluble. This change is only partially reversible when the hide is subsequently soaked in water as in the case of milling under the experimental conditions described earlier.

While the hides are in stack those halophylic bacteria which are still active will break down the protein matter, and in particular the globular proteins which are more susceptible to attack, thereby rendering them more readily soluble in the soak liquor. Putrefaction is, of course, the main danger in the curing of hides and skins, hence a study of the breakdown products of proteins will give information as to the efficiency of the cure.

Sodium fluosilicate is a lyotropic or hydrotropic agent, i.e. it has the ability to disperse native or denatured globular proteins, consequently some of these materials will be extractable by water. Actually in the milling method used the salt from the hide cubes will form a dilute (approximately 3 per cent) brine which is more efficient for such extraction than pure water.

Two points should be borne in mind when examining the figures for water-extractable and volatile nitrogen shown in the Tables and in Fig. 5 and 6. In the first place a three-hour soak as applied here is not sufficient to extract all the nitrogen available, and only the more readily soluble particles will be accounted for. Secondly, the amount of nitrogenous matter extractable depends upon the amount available, i. e. where large amounts have been removed during the actual curing process (see above) the amount extracted in the soak will be less. Volatile nitrogen is an indication of bacterial activity, the amount present in the soak depending upon the efficiency of curing. The three-hour soak is sufficient to bring the volatile nitrogen to equilibrium and the values shown in the Tables can therefore be taken as the total amount of residual volatile nitrogen. A certain amount of this nitrogen will, of course, dissipate into the air during curing and cannot be determined. Where sodium fluosilicate is used, however, this acid salt will retain the volatile bases, so that the values in this case will be fairly accurate.

Fig. 5 shows the amount of extractable and volatile nitrogen (on the same scale) as a function of the length of curing. It is at once apparent that sodium fluosilicate is an excellent bactericide, as evidenced by the fact that no matter what amount of salt is used or how long the curing period the volatile nitrogen stays constant. This can only mean that no putrefaction could take place in the hides under any of the curing conditions employed in this work. In the control hides the increase in volatile nitrogen with increased length of curing periods indicates bacterial activity, and it is interesting to note that the rate remains more or less constant from 10 to 90 days.

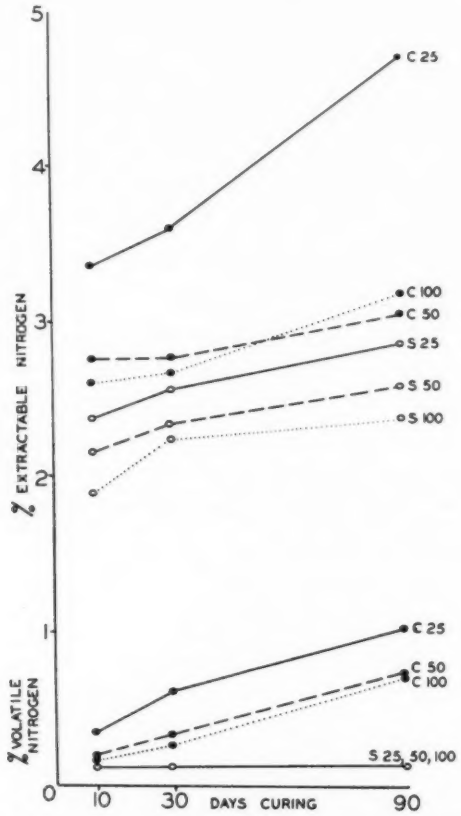

FIGURE 5.—Influence of the curing period on the amount of extractable and volatile nitrogen in wet-salted hides, using different amounts of salt.

The values for extractable nitrogen are also very interesting. In the controls there is a more pronounced increase with increasing length of curing than with the fluosilicate hides. This is especially true of the control group which had received only 25 per cent salt. In the fluosilicate hides no bacterial action could have been responsible for increased solubility of globular protein material, therefore the increase in this case must be ascribed to the dispersive effects of the sodium fluosilicate upon the globular proteins. In the controls the increase must be due to bacterial breakdown of the protein whereby it becomes more readily soluble. This is proved by the fact that the solubilization of protein matter parallels the development of volatile nitrogen.

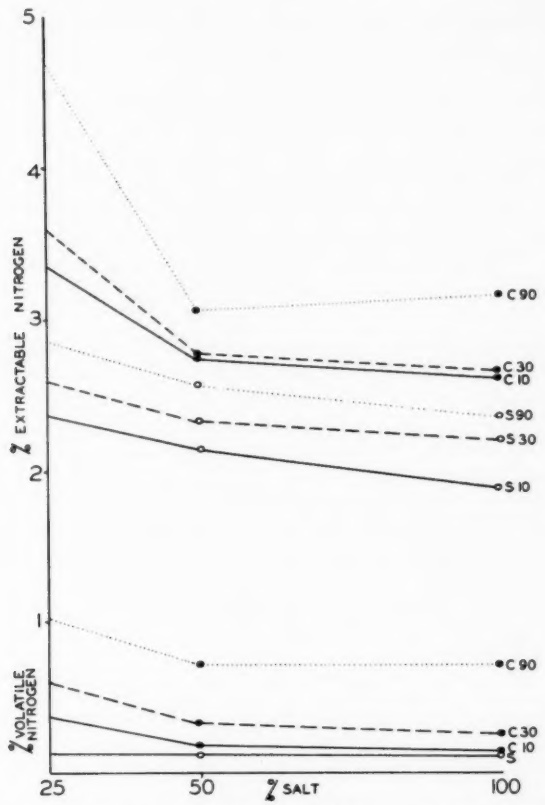

FIGURE 6.—Influence of the amount of salt on the amount of extractable and volatile nitrogen in wet-salted hides cured for varying periods.

Fig. 6 shows the influence of the amount of salt applied on the extractable and volatile nitrogen. From this Figure it is clear that volatile nitrogen development, respectively bacterial activity, is dependent upon the amount of salt used in the control hides. There is little difference between the values at 50 per cent and at 100 per cent salt, but 25 per cent salt obviously allows of considerable breakdown of the protein. Where sodium fluosilicate had been used the amount of salt applied is immaterial insofar as volatile nitrogen development is concerned.

The values for extractable nitrogen present a more complicated picture. Taking into account the fact that bacterial activity in the control hides will result in more nitrogen being extractable in the soak, it might be thought

that the decrease in the amount extractable with increasing amounts of salt is only due to the higher efficiency of large amounts of salt in preventing bacterial breakdown. Partly this is true, of course. But in the sodium fluosilicate series, where no such bacterial breakdown could have occurred, there is also a decrease in the amount of nitrogen extracted with increasing amounts of salt applied to the hide. The difference is especially apparent between 25 per cent and 50 per cent salt. In these hides the decrease can only be due to changes in the protein brought about by the increased amounts of salt. The explanation lies in the fact that large amounts of salt will more easily form a concentrated brine, thereby salting out the proteins more effectively and to a greater extent. Their removal in the soak is then more difficult.

Since removal of globular proteins is a desirable feature of the beamhouse process, it would appear that the best results would be obtained with hides which had received a small amount of salt (e. g. 25 per cent) for a long period (e.g. 90 days). Under these conditions bacterial action will, however, damage the valuable proteins of the hide also. Taking the view that the stock would be of the best quality if it shows a low volatile nitrogen content, then it would seem that not less than 50 per cent salt should be used for a period of not more than 10 days. If sodium fluosilicate is used in the curing process then the amount of salt and the length of the curing period have no influence on the development of volatile nitrogen (which remains at a minimum), but again 25 per cent salt for 90 days would result in the best removal of globular protein material.

The Limed Yield

Limed yield, as measured in these experiments, is an indication of the degree of rehydration of the stock in the soak and lime liquors. Both primary swelling (in the soak pits) and secondary swelling (in the alkaline lime pits) depend upon the ease with which water can reach the various components, especially the fibres, and the space available to such fibres to swell. Finally, in secondary swelling, the degree of splitting up of the fibres is concerned.

A number of factors can therefore influence the swelling. In the case of wet-salted hides these are: the salt present, the degree of denaturation of the interfibrillar proteins, the degree of denaturation of the fibers themselves, the degree of splitting or breakdown of the reticular fibres which hold the collagen fibres in bundles.

In the experimental hides used here the degree of denaturation of the collagen would be more or less uniform for all the hides, and need therefore not be considered.

The amount of salt present and the denaturation of the globular proteins are two factors which are related. The more salt and the quicker it penetrates the hide to form a concentrated brine, the more the globular proteins

are precipitated and denatured. It will then be more difficult to remove such globular proteins in the process and they will retard proper penetration of the water into the interfibrillar spaces and proper swelling of the fibres. Reticular fibres are broken down to some extent by sodium chloride, hence their condition will also depend upon the salt content and in turn they will influence the splitting of collagen fibre bundles.

When considering limed yield on the cured weight several more factors come into play, all of which can be referred back to the shrinkage values obtained during curing. The higher the shrinkage value, the more the stock has dried out during curing, therefore it can take up more moisture in soaking and liming. The reverse also holds true.

The limed yields measured here were taken after a light fleshing in the limed state, to conform to normal tannery practice. For this reason the limed yield on green weight does not reach the expected values of more than 100 per cent. The method has, nevertheless, been standardized at the tannery in question and the yields are comparable with one another.

The principles outlined above are borne out by the results depicted in Fig. 7 and 8. In Fig. 7 the limed yield has been plotted against the curing period and in Fig. 8 against the amount of salt used. Two sets of curves are shown in each Figure: one represents the limed yield on green weight and the other the limed yield on tannery weight (i.e. the final cured weight). By comparison with Fig. 1 and 2 it will immediately be obvious that there is a distinct correlation between shrinkage and the limed yield on cured weight. The correlation between limed yield and green weight is less distinct.

The length of the curing period affects the limed yield on cured weight (Fig. 7). This is largely due to the fact that with increased curing there is increased loss of moisture, hence the stock will take up more moisture in the beamhouse. Other factors in this connection will be discussed below. Similarly the decrease in the limed yield as a function of increasing amounts of salt used (Fig. 8) is largely due to the decreased shrinkage discussed earlier in detail.

The limed yield on green weight shows the same trends as that on cured weight, although less distinctly. Here the influence of time is probably two-fold, but it is difficult to tell which is the more important. In the first place, increased length of storage will result in increased breakdown of the reticular tissue, thus enabling the collagen fibres to split more. The limed yield will then increase. Secondly, in the control hides bacteria will break down the interfibrillar proteins to render their removal more easy, with the result that the fibres are not unduly prevented from swelling. The sodium fluosilicate will disperse these globular proteins and the same net result is obtained.

The amount of salt also influences the limed yield on green weight in the case of the control hides. The correlation only holds as long as less than

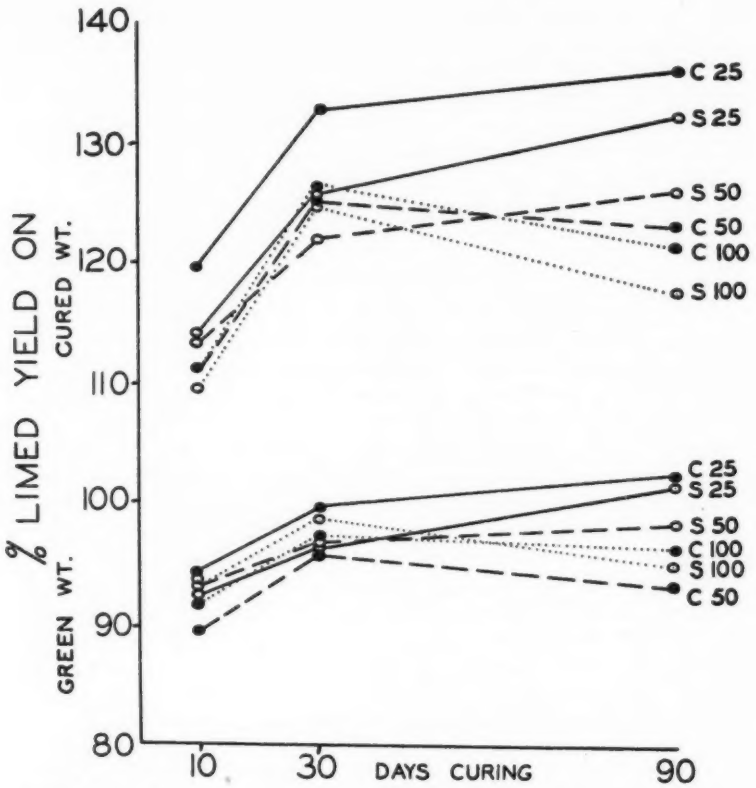

FIGURE 7.—Influence on the curing period on the limed yield (on cured and green basis) from wet-salted hides, using different amounts of salt.

50 per cent salt is used. In the sodium fluosilicate hides the amount of salt does not affect the limed yield on green weight, therfore, it seems clear that bacterial action is responsible for the high limed yields in the control hides receiving only 25 per cent. Larger amounts of salt effectively prevent such bacterial action (see Fig. 6) which would otherwise affect the globular protein in such a way that they can be removed quite easily.

In these limed yield studies the length of curing and the amount of salt again appear as two opposing factors. The highest limed yields are obtained with the least amount of salt and the longest period of curing, and vice versa.

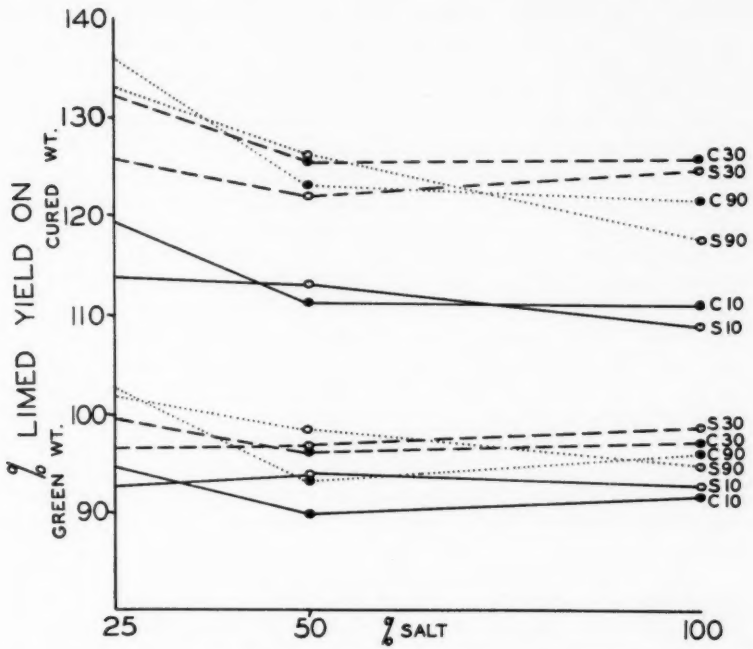

FIGURE 8.—Influence of the amount of salt on the limed yield (on cured and green basis) from wet-salted hides cured for varying periods.

The Optimum Curing Conditions

From what has been said above it is clear that the interests of the tanner and of the curer clash in many respects, but there is also contradiction in the requirements of a cure which will give the best results.

On the basis of shrinkage the curer would prefer to use excess salt for as short a time as possible in order to lose as little weight as possible. The tanner would, of course, prefer to buy a higher percentage of actual hide substance, i. e. he wants a long cure with little salt used. This factor is purely commercial. Taking the removal of globular proteins as a criterion of quality a small amount of salt and long curing period would be required. On the other hand, if the amount of volatile nitrogen evolved is to be considered, then a large amount of salt and a short curing period would be desirable. Finally, since the concentration of sodium chloride in the residual moisture determines the curing efficiency, an excess of salt and a curing period of at least ten days are necessary.

In considering the cure which would best fit the varying requirements and interests, it is necessary to remember that the prime purpose of curing is to present the tanner with material which would resemble the fresh as closely as possible when the soaking process is completed. This requires, basically, that bacterial damage should be avoided or at least minimized. It can be achieved by saturating the residual moisture inside the hide as quickly as possible with sodium chloride, i. e. by using not less than 50 per cent salt along commercial practice lines and keeping the hides in stack for about ten days. If sodium fluosilicate is used with the salt the hides may be kept for any length of time and much less salt need be used, without affecting the inherent quality of the goods. The question of weight losses will, of course, force the curer to use sufficient salt even where sodium fluosilicate is applied.

A second, much less important purpose of curing is to remove the undesirable globular proteins, or at least not to render their subsequent removal in the beamhouse more difficult. When using salt only this requires a small amount of salt and long period of curing as shown before, but such removal depends upon bacterial action which will also harm the fibrous proteins. A high volatile nitrogen content must be avoided. Here sodium fluosilicate may again be used with good effect, since it does not prevent the subsequent removal of the interfibrillar proteins and actually assists this process by its dispersive action.

A third factor in curing is the breakdown of reticular tissue, whereby the collagen fibres are allowed to split up. This action merely requires time, but is not of sufficient importance to endanger the proteins unnecessarily by excessively long curing.

It is clear from this discussion that the best curing, i. e. preservation of the valuable collagen, can be achieved by using about 50 per cent salt on hide weight, the salt containing 2 per cent sodium fluosilicate, and storing the hides for about ten days.

This cure would give low shrinkage values, in other words the tanner would buy relatively less hide substance. It is however a matter for agreement between tanner and curer and does not concern the efficiency of preventing bacterial damage to the stock. Perhaps a cure which would satisfy both curers and tanners without decreasing its efficiency would be to use about 50 per cent salt, mixed with sodium fluosilicate, and storing the hides for 30 days.

Summary

In a detailed study of various factors concerned in the wet-salting of heavy abattoir hides the following information has been obtained:

1. Shrinkage from the green to the cured weight is a function of the amount of salt used and the length of the curing period: the less salt and the longer the curing, the higher is the shrinkage. Values ranged from 25 per cent to 15 per cent in these experiments.

2. Addition of 2 per cent sodium fluosilicate to the curing salt causes a decrease of about 2 per cent in the shrinkage values.

3. If only 25 per cent salt on green weight is used the residual moisture in the hide will not be saturated with sodium chloride even after 90 days of curing. The extra moisture retained where sodium fluosilicate is used is also saturated provided sufficient salt is applied.

4. The amount of total nitrogen extractable by milling hide cubes in water for three hours increases with a decrease in the amount of salt used and with an increase in the length of the curing period.

5. Volatile nitrogen development parallels the amount of total extractable nitrogen, except when sodium fluosilicate is used. In this case the volatile nitrogen content remains at a minimum level, no matter how much salt is used or how long the curing period.

6. Limed yield on cured weight is directly proportional to the amount of shrinkage occurring during curing.

7. Limed yield on green weight increases with decreasing amounts of salt and increasing length of curing. Where sodium fluosilicate is used the amount of salt used does not affect this limed yield.

8. Quite efficient curing of wet-salted hides can be obtained by applying about 50 per cent salt, containing 2 per cent sodium fluosilicate, on green weight and storing the stock for about ten days.

Leather Industries Research Institute,
Grahamstown, South Africa.
30th, November, 1950.

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BOOK NOTICES

ORGANIC CHEMISTRY. By E. Wertheim. Third Edition. The Blakiston Company, New York. XII plus 958 pages. 1951. Price $5.00.

The book is primarily a textbook for a course in elementary organic chemistry, and is excellent for the purpose. Divided into two main divisions, aliphatic and aromatic, each division is broken up into natural subdivisions, logically arranged. The helpful devices known to the teaching profession are freely employed. For example, hints on how to study

are given, important words of nomenclature are emphasized with heavy print, and charts, showing the chemical relationships and reactions of the substances chosen for basic study are given. Review questions, colored plates, numerous figures and tables, rather extensive bibliographies and short biographies with photographs of outstanding organic chemists are further examples.

The text is easy reading and clear. Subjects of general public interest are well selected. For instance, the student will get a good idea of what antihistamine means, what antihistamines are, how they work and why they are needed. Tanning chemistry, not being a subject for the elementary student, is just one of the interesting interpolations.

The book is obviously not intended to be a reference work for the advanced organic student or specialist in the field. However the very qualifications, which make it a good student text, also make it valuable for those who wish to brush up, or desire a reference giving readily accessible and simple explanations of the fundamentals. The bibliographies make it easy to expand the study if desired. R. H. T.

ABSTRACTS

The Hydration of Amino Acids. By Y. Wada, S. Shimbo, M. Oda, J. Nagumo, and K. Shimura. *J. Agr. Chem. Soc., Japan*, **23,** 258 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 8203 1950.

The apparent molal volume of amino acids in aqueous solutions increases with the ionic strength of other simple ions. From the fact that the relation between molal volume and ionic strength was equal for 4 amino acids with different hydrocarbon parts (glycine, alanine, α-amino-*n*-butyric acid, and valine), the hydration of hydrocarbon part was concluded not to be accompanied by electrostriction. Two kinds of hydration were expected, first, due to dissociative radicals, $-NH_3^+$ and $-COO^-$, and, second, due to the hydrocarbon part.

Electrophoresis of Proteins in Filter Paper. By F. Turba and H. J. Enenkel. *Naturwissenschaften*, **37,** 93 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 8407 (1950).

A strip of filter paper (Whatman No. 1 or 4, Schleicher and Schüll No. 598g, etc.) 5 X 25 cm. was stretched out in a moist chamber, soaked in buffer solution and the ends dipped in the same. In the middle 1 to 3 drops (0.03 to 0.1 cc.) of a protein solution were deposited and 110 v. d. c. applied. The proteins separated electrophoretically as shown by placing the strip in a dye solution (50 per cent) aqueous methyl alcohol, 10 per cent acetic acid, cold saturated with azocarmine) for 10 minutes, then 5 minutes in methyl alcohol and 5 minutes in 10 per cent acetic acid. The protein spots become intensive red to deep purple. With serum protein, 0.1 molar veronal buffer (pH 8.6), 12 to 24 hours, (1.0 to 2.0 ma.) separation into one albumin and three globulin fractions was found (photographs). Quantitative evaluation can be done by retentiography [Wieland, *Chem. Abstracts, A. C. S.*, **43,** 2660c (1949)] or by elution of the pigment after cutting the strip and colorimetry. Pathological serums show the expected abnormalities (nephrosis serum). The method may be of clinical value. For preparation purposes a column of buffer-soaked filter paper disks is used.

Dermatological Hazards in the Leather Industry. By M. H. Samitz. *Ind. Med.*, **17,** 431 (1948). Through *Chem. Abstracts, A. C. S.*, **44,** 8687 (1950).

Leather tanning is described. The causative agents responsible for dermatitides and the types of eruptions prone to occur in this industry are reviewed, with an outline of preventive measures.

Utilization of the By-Products of Plant Material Extraction. Ital. Pat. 415, 982. A. B. I. Azienda Brevetti Industriali Societa a R. L. Apl. Nov. 12, 1946. Through *Chem. Abstracts, A. C. S.*, **44,** 8687 (1950).

The process deals with the further treatment of the comminuted plant materials used in the tannin industry (chestnut, etc.) after the extraction with hot water is over. It consists in cooking the residue in an autoclave with 5 times its weight of a 2 per cent H_2SO_4 solution for less than 6 hours, and at a temperature between 120° and 200°C. for the extraction of pentosans and other sugary material for the preparation of furfurol. The new residue, after washing, is subjected to mechanical disintegration of the fibers and further successive filterings under pressure at temperature between 120° and 200°C. for separation of solids of various specific gravities used for insulation and other purposes.

Quantitative Analysis of Mixtures of Two Weak Acids by Conductometric Titration. By J. Schleicher and L. Pivet. *Bol. Soc. Chilena Quim.*, **1,** 9 (1949). Through *Chem. Abstracts, A. C. S.*, **44,** 8815 (1950).

The change in the electrometric conduction of 100 ml. portions of 0.5 to 0.01 *N* solutions of acetic acid, tartaric and oxalic acid upon gradual addition of 10 ml. of NaOH solution of 10 times the normality of the acid solutions was measured, also that for mixtures of equal volumes of pairs of these acid solutions, for mixtures of pair, of the 0.1 *N* acids in varying proportions, and for some other single acids of varying dissociation constants. The results are shown in tables and graphs. The following formulas have been computed for the relation between *N* and *t*, where *t* is the percentage fraction of total acid neutralized at the point of minimum cond.: acetic acid, $t=0.5225 \times N^{-0.5213}$ for 0.5-0.0035 *N* and $t=3$-0.75; tartaric acid: $t=3.308 \times N^{-0.5368}$ for 0.5-0.035 *N* and $t=20$-4.8-; oxalic acid: $t=54.64$-23.2 *N* for 0.01-0.2 *N* and $t=50$-54.5. Similar formulas are given for mixtures of two acids. For seven weak acids studied *t* increases with the first dissociation constant. The minimum conduction of mixtures of two weak acids is always between the minimum values for the single acids of equal normality. Unknown mixtures can be analyzed satisfactorily if the first dissociation constants are between 10^{-2} and 10^{-6}; the precision increases with the difference between the dissociation constants of the two acids.

Elimination of Losses in the Estimation of Amino Acids by Paper Chromatography. By L. Fowden and J. R. Penney. *Nature*, **165,** 846 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 8826 (1950).

Recovery of amino acids after chromatographic separation on paper strips with PhOH-NH_4OH as solvent, drying at 75°-100°C. and colorimetric determination with ninhydrin is usually 75-80 per cent. Recovery with butyric acid-acetic acid solvent is somewhat higher. Experiments show that drying the paper at high temperature in the presence of solvent causes the losses of amino acids. Removal of the solvent with diethyl ether, drying at room temperature and subsequent color development resulted in 98 to 101 per cent recovery of alanine, phenylalanine, and glutamic acid; phenylalanine gives only 60 per cent recovery when dried in the presence of solvent. By elimination of the hot-air drying stage losses on two dimensional chromatograms were not greater than 8 per cent.

Deamination of Amino Acids by Peroxidase. By M. Solsona and M. Mora. *Rev. Espan. Fisiol.*, **5,** 281 (1949). Through *Chem. Abstracts, A. C. S.*, **44,** 8977 (1950).

Neither H_2O_2 nor H_2O_2 with a peroxidase derived from homoglobin was able to deaminate the amino acids—glycine, alanine, leucine, glutamic acid, and aspartic acid. Ammonia was split off from asparagine, this ammonia probably being of amide origin since the $-NH_2$ group in asparatic acid was not affected by the treatment. The acids and the asparagine were

heated in acid solution (H_2SO_4 used) with H_2O_2 for 24 hours at 35°C. Deamination also did not occur when amino acids were treated in an alkaline medium with 3 per cent H_2O_2 or 1.5 per cent H_2O_2. Asparagine, however, yielded ammonia.

Titrations in Nonaqueous Solutions. 1. Bromometry in Glacial Acetic Acid and Determination of Iodine Number by a Direct Titration with Bromine. By O. Tomiček and J. Doležal. *Chem. Listy*, **43,** 193 (1949). Through *Chem. Abstracts, A. C. S.*, **44,** 9297 (1950).

Organic compounds were successfully titrated with bromine solution in glacial acetic acid. The indicator electrode was a platinum spiral, the reference electrode a normal calomel electrode. Titrations were carried out at 20° and 60C°. If the direction titration did not work, excess Br_2 was added and back-titrated with arsenite solution. Saturated solution of sodium acetate in acetic acid was used as a buffer. Phenol, *l*-naphthol, thymol, pyrocatechol, cholesterol, limonene, naphthalene, and oleic acid were determined. The method was applied to the determination of unsaturated compounds in oils and essential oils (cottonseed, olive, tape, linseed, and cod oils, citronella, peppermint, rosemary, and cedar essential oils).

Colorimetric Determination of Calcium by Means of Pyrogallolcarboxylic Acid. By J. Pech. *Chem. Listy*, **43,** 8 (1949). Through *Chem. Abstracts, A. C. S.*, **44,** 9298 (1950).

Pyrogallolcarboxylic acid gives with calcium a blue-violet coloration in alkaline solutions. For the quantitative colorimetric measurements, a filter S 57 and the width of 0.5 cm. were found the most suitable. Concentration of 0.2-0.4 mg: Calcium in 10 ml. of test solution were used. For higher concentrations of calcium, a protective colloid (2 per cent starch solution) must be used to make the extinction curve linear. To 10 ml. of a solution containing 0.2-0.4 mg. calcium add 1 ml. 2 *N* NaOH, 2 ml. 2 per cent starch solution, and dilute with a saturated aqueous solution of pyrogallolcarboxylic acid to 50 ml.

The Bromometric Determination of Formic Acid. By W. Poethke. *Pharm. Zentralhalle*, **86,** 357 (1947); *Chem. Zentr.*, **I,** 1093 (1948). Through *Chem. Abstracts, A. C. S.*, **44,** 9303 (1950).

A modification of the method of Mäder is reported. To accelerate the reaction between BrO_3^- and Br^- the mixture is acidified and then neutralized with sodium acetate. The following procedure is given: Place the HCOOH or formate solution in a 250 cc. long-necked volumetric flask. Add enough 0.1 *N* $KBrO_3$ solution to give an excess of at least 10 cc. Then add 3-4 g. KBr and dilute the mixture with water to about 80 cc. After solution is complete, acidify with 6.5 cc. 12.5 per cent HCl and close the flask tightly. After 2 minutes, neutralize the mixture with 12 cc. 2 *N* sodium acetate and place the tightly stoppered flask in the dark for 30 minutes. Then add 1 g. of KI dissolved in 5 cc. water and 6 cc. HCl (12.5 per cent) and titrate the liberated iodine with 0.1 *N* $Na_2S_2O_3$.

Molecular Structure of α-keratin. By S. Mizushima and T. Shimanouchi. *Repts. Radiation Chem. Research Inst., Tokyo Univ.*, **5,** 4 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 9495 (1950).

The molecular structure of α-keratin proposed previously by the authors [*Chem. Abstracts, A. C. S.*, **44,** 2043 (1950)] is also supported by the results of the near infrared measurements made on α-keratin and related compounds.

Separation of Amino Acids and Peptides by Chromatographic Method. II. Effect of Hydrochloric Acid on Paper Chromatograms of Amino Acids. By S. Ishii and T. Ando. *Repts. Radiation Chem. Research Inst., Tokyo Univ.*, **5**, 53 (1950). Through *Chem. Abstracts, A. C. S.*, **44**, 9503 (1950).

The effect of HCl, contained in the sample to be analyzed, on the paper chromatograms of amino acids was studied in different solvents. With increasing concentration of HCl the R_f value of an amino acid increased in butanol and decreased in isovalerianic acid. In phenol and in collidine-lutidine such an effect was only found with a few amino acids.

Waste Waters from Tanneries, Dairies and Metalpickling Plants. By E. Leclerc and I. Beeckmans. *Bull. Centre Belge Étude et Document. Eaux Liége)* 279, 353 (1949). Through *Chem. Abstracts, A. C. S.*, **44**, 9599 (1950).

A review with 62 references.

Treatment of Meat-Packing Wastes. By L. Bradney, W. Nelson and R. E. Bragstad. *Sewage and Ind. Wastes*, **22**, 807 (1950). Through *Chem. Abstracts, A. C. S.*, **44**, 9599 (1950).

The sewage consists of half domestic sewage and half packing-house wastes. Grease and high nitrogen content are the most difficult problems. These should be removed as much as possible at the packing plant. Paunch manure is removed by fine screens at the packing plant. The sewage-treatment plant consists of primary sedimentation, separate sludge digestion, roughing filters of the back-wash type loaded at 1000-15,000 pounds B. O. D./acre ft., followed by standard filters with recirculation of about 1:1 and a loading up to 12,000 pounds B. O. D./acre ft., and standard filters followed by the activated sludge process loaded up to 50 pounds B. O. D./1000 cu. ft. aeration capacity. The chief difficulty in operation has been due to rising sludge in the clarifiers which follows both the standard filters and the activated sludge process. This is caused by high production of nitrites and nitrates which are reduced in the summer months to gaseous N_2. The problem was finally solved by rapid removal of the sludge from the clarifiers by suction-type scrapers. These had to be modified with V-shaped scrapers between the suction nozzles and adjustments of orifice sizes in the suction nozzles. These changes together with evaporation of stick liquor by the industry during peak winter loads have produced a final plant effluent of 8-20 p.p.m. B. O. D. during 1947.

Pyrogallolcarboxylic Acid as a Reagent for Determining Calcium. By A. Okáč and J. Pech. *Chem. Listy*, **42**, 161, (1948). Through *Chem. Abstracts, A. C. S.*, **44**, 9857 (1950).

A saturated aqueous solution of 2, 3, 4-pyrogallol-1-carboxylic acid gives a blue-violet coloration with Ba^{++}, Sr^{++} and Ca^{++}. The reagent can be used for detecting Ca^{++} in the presence of Ba^{++} and Sr^{++}, if the solution is treated with a slight excess of 9 *N* H_2SO_4, boiled, cooled, and filtered before adding the organic reagent. The test can be used for the colorimetric determination of calcium in drinking water.

New, Very Sensitive Color Reactions of Magnesium. By J. Knop. *Chem. Listy*, **41**, 273 (1947). Through *Chem. Abstracts, A. C. S.*, **44**, 9862 (1950).

Eriochrome Gray SGL and BL (Geigy, Switzerland) show in the presence of very small accounts of magnesium a rose to purple coloration. A 0.1 per cent solution of the dye is used as the reagent which has a blue color. To a sample containing magnesium add 0.05 ml. 0.1 per cent solution of the dye and 0.1 ml. 10 per cent NH_4OH. Ag, Sn, As, Sb, Na, K, and Li do not interfere. Cu, Cd, Zn, Hg, and Co must be masked. Mn and Fe interfere.

Conductometric Studies on Complex Amino Salts. By C. Gensch. *Z. Anal. Chem.* **131,** 89 (1950). Through *Chem. Abstracts, A. C. S.* **44,** 9870 (1950).

The NH_3 content in ammonium salts can be determined by conductometric titration with NaOH. The purpose of the present work was to determine the NH_3 content in complexes and, if possible, the simultaneous determination of the metal ion serving as the central atom. The Cu ammoniates gave only the approximate NH_3 content and the values obtained for the Cu content were worthless. With $[Ni(NH_3)_6]^{2+}$ salts, after addition of HCl and back titration with NaOH, good values were obtained. $Ag(NH_3)_2NO_3$, in contrast to the corresponding Cu and Ni salts, gives a clear solution with water so that it was thought that the nearly neutral solution could be titrated directly without a preliminary treatment with acid. Direct titration with NaOH gave results which were not satisfactory for either the NH_3 or the Ag but back titration gave good results. In the case of $Zn(NH_3)_4Cl_2$ containing a little excess acid, the titration values were good both for Zn and for NH_3. Cd ammines, contrary to expectations, did not behave as well. On the other hand $[Co(NH_3)_5Cl]\cdot Cl_2$ and $[Cr(NH_3)_6](NO_3)_3$ conductometrically titrated with NaOH gave good results. The analysis of $HG(NH_3)_6Cl_2$ gave good results for NH_3 after preliminary treatments with acid but the determination of the Hg was unsuccessful.

Swelling of Collagen in Alkaline Solutions. II. Swelling in Solutions of Univalen Bases. By J. H. Bowes and R. H. Kenten. *Biochem. J.*, **46,** 524 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 10,010 (1950).

Differences in swelling of oxhide collagen in basic solutions up to pH 12.5 are supposed to be due to variations in affinity of the cations for collagen. Comparison of swelling curves obtained with NaOH and with guanidine or $(CH_3)_4NOH$ indicate that the increased swelling at high pH values is due to the increased concentration of OH and not to an effect of Na ions. Organic solvents presumably decrease the swelling pressure rather than increase cohesion. The view is developed that the decrease in cohesion of collagen at high pH is associated with the breaking of intermolecular linkages between the polypeptide mols. This may involve not only H bonds but also NH_2 groups. Thus, it was found that soaking collagen in NaOH of pH 13.7 for 3 days decreased the amide N by two-thirds and caused over 700 per cent swelling. On the contrary, treatment with NH_4OH or diethylamine of the same pH caused no swelling or decrease in N.

Swelling in Solutions of Bivalent Bases. III. By J. H. Bowes. *Biochem. J.*, **46,** 530 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 10,010 (1950).

Swelling of collagen in solutions of bivalent bases decreases as follows: $Ba(OH)_2 \geqslant Sr(OH)_2 > Ca(OH)_2$. The small degree of swelling in $Ca(OH)_2$ is probably due to combination of calcium ions with collagen. The swelling effect of $Ba(OH)_2$ is less than one-half of NaOH; that of $Ca(OH)_2$ is only about one-eighth. Addition of $CaCl_2$ or NaCl to the $Ca(OH)_2$ solution increases the swelling, in the former case perhaps because of decreased cohesion of the collagen and in the latter because of increase in swelling pressure.

Measurement of Reducing Sugars by Ferricyanide in Alkaline Medium. By M. Plumel. *Bull. Soc. Chim. Biol.*, **31,** 1163 (1949). Through *Chem. Abstracts, A. C. S.*, **44,** 10,027 (1950).

The influence of alkali concentration, time of heating, and concentration of oxidizing agent on the oxidation of glucose by ferricyanide were studied in order to obtain perfect proportionality between the amount of glucose and the oxidizing agent consumed. The method of Hagedorn and Jensen [*Chem. Abstracts, A. C. S.*, **17,** 2721 (1923)] was used on

0.1 cc. samples of blood and 0.2 cc. of spinal fluid. Proteins were precipitated with $CdSO_4$ and NaOH according to the method of Fujita and Iwatake [*Chem. Abstracts, A. C. S.*, **26,** 1313 (1932)]. This method was compared to Folin's microcolorimetric method, based on the amount of ferrocyanide formed by precipitation as Prussian blue, after precipitation of proteins by $Fe(OH)_3$.

Distinction between Ouricury Wax and Carnauba Wax. By C. Ludecke and M. Diena. *Ind. Vernice* (Milan), **2,** 52 (1948). Through *Chem. Abstracts, A. C. S.*, **44,** 10,355 (1950).

Ouricury wax can be distinguished from carnauba wax (a) by the red coloration which it gives on heating 1 g. with 10 g. of 25 per cent KOH solution, the wax becoming red but the aqueous layer remaining colorless; carnauba remains colorless but colors the aqueous layer yellow; (b) by the difference between its melting point and its solidification point (melting point of refined ouricury wax 80°-82°C., solidification point 71°C.). Other constituents of the wax are reported *(loc. cit.)*.

Polarographic Studies in Chemistry of Tannins. VI. Polarograms of Oxidized Split Products of Tannins in Their Aqueous Solutions. By A. K. Vlček, E. Špalek, L. Krátký, and E. Havlíček. *Chem. Obzor*, **25,** 81 (1959). Through *Chem. Abstracts, A. C. S.*, **44,** 11,147 (1950).

Pyrocatechol, resorcinol, phloroglucinol, pyrogallol, and gallic acid were investigated. After 8 hours' oxidation by air, at pH 4 to 10.4, in solutions of pyrocatechol, phloroglucinol and resorcinol, no cathodic polarographic waves were observed. This was contrary to findings of others. In aqueous solutions of pyrogallol, oxidized by air, two polarographic cathodic waves arise. One of these waves is well developed and its $\pi^{1/2}$ varies from -770 mv. to -590 mv. at pH range of 1.8 to 8.0. The second wave is gradually ascending and its $\pi^{1/2}$ varies from -580 mv. to -590 mv. at the same range of pH. The first wave pertains to quinone, an intermediate oxidation product and the second wave to purpurogallin, the second oxidation product. The height of the cathodic wave pertaining to quinone first increases as the oxidation increases, then its increase diminishes and finally declines so that this wave fuses into the polarographic wave of the final oxidation product, the height of which rises during the oxidation. With the gallic acid, after the oxidation by air, at pH range of 2.0 to 9.5, only a single wave arises of $\pi^{1/2}$ from -620 mv. to -780 mv., as at once the intermediate quinone product continues to oxidize. The waves of pyrogallol easily arise, even at the oxidation by air in a neutral medium and their heights increase if the oxidation takes place at higher temperature. The rise of cathodic wave in a aqueous solution of gallic acid requires at room temperature an increased oxidation and reaction at higher pH. At 60°C. a cathodic wave arises even at the oxidation in neutral solutions. The waves of pyrogallol and gallic acid may easily occur in the aqueous extracts of natural tannins. [Cf. *Chem. Abstracts, A. C. S.*, **40,** 6279 (1946)].

Actual State of the Question of Methods of Analysis. By G. A. Bravo. *Cuoio, Pelli. Hat. Concianti*, **26,** 46 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 11,149 (1950).

After the dissolution of the International Society of Leather Trades' Chemists, every national association shall define the official methods for the analysis of tannins, leather, etc. The Italian Association of Leather Chemists has approved the Filter Method as official for the analysis of tannins (only for the home trade). The question of the methods of leather analysis is not yet defined.

Comparative Analytical Data on Shoe Sole (Leather) Divided into Strata. By A. A. Giacomi. *Rev. Facultad cienc. Quim.*, **22,** 155 (1947). (Pub. 1949). Through *Chem. Abstracts, A. C. S.*, **44,** 11,149 (1950).

Sixteen samples of sole leather (of which 11 were tanned with highly sulfited quebracho extracts, two with mixed vegetable tannins, one according to Wilson [*Modern Practice in Leather Manufacture*, **1941** *(Chem. Abstracts, A. C. S.*, **35,** 3585 (1941)], and two of unknown origin) were cut into four strata (from the grain to the flesh side) 0.5 to 1 mm. thick, and a representative central section was cut into a number of panels which were subjected to chemical analysis and to physical testing (12 samples only). The chemical tests which comprised percentage of moisture, percentage of fatty matter, percentage of leather substance, percentage of insoluble ash, percentage of combined tannin, percentage of hide substance, degree of tanning (by the 1937 methods of the American Leather Chemists' Association), and percentage of soluble matter (by the 1938 methods of the International Society of Leather Trades' Chemists) disclosed no substantial difference between Argentina and foreign hides except the lower degree of tanning in 11 of the former. Physical tests which included thickness, apparent density, tear resistance, water absorption, wear (abrasion) resistance, and resistance to cracking, could not be correlated with the chemical composition. The inferior degree of tanning (even though widely different tanning periods were applied) is thought to be caused by the lack of fixation of the sulfited quebracho in spite of good penetration. Also, the fat content of the hides was generally low. Analysis of hide strata did not disclose any important data not obtainable by analyzing the whole hide. The Argentina hides were lower in wear resistance, slightly thinner, and less dense than the foreign hides tested.

Electron Microscope Study of Collagen. By A. L. Zaides and S. L. Pupko. *Kolloid, Zuhr*, **12,** 275 (1950). Through *Chem. Abstracts, A. C. S.*, **44,** 11,150 (1950).

Collagen fibers of cowhide consist of alternate dark and light disks; in untreated collagen they are, respectively, 560 and 300 A. thick, but the thickness is reduced by tanning, e.g., to 440 and 260 A. after treatment with oak extract, to 435 and 215 A. by $UO_2(OAc)_2$, and to 340 and 210 A. by phosphotungstic acid. In treated collagen, there are also dark layers, on the average 80 A. thick, and light layers of 60 A.; this periodicity must be due to orientated adhesion of heavy metal. A fiber ruptures always within a light disk. After a fiber is broken, the light disks remain elongated, while the dark disks contract and assume the shape of a drop. This shows that the polypeptide chains are not folded in the dark disks.

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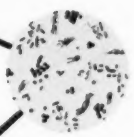

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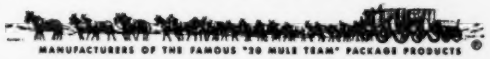

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